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- FINAL TECHNICAL REPORT -

**DEVELOPMENT OF MERCURIC IODIDE**

**UNCOOLED X-RAY DETECTORS AND SPECTROMETERS**

**FOR SPACE MISSIONS**

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For the Period Ending September 30, 1989  
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## I. INTRODUCTION

This report summarizes the results which were obtained under an ongoing, multiyear program to develop miniature, low power, light weight mercuric iodide ( $HgI_2$ ) x-ray spectrometers for future NASA space missions. The developments reported here are for the period ending September 30, 1989 (which includes a three month no-cost extension period requested by the Principal Investigator).

In earlier progress reports we have reported on the potential advantages, in space applications, that might be derived from the use of  $HgI_2$  x-ray fluorescence spectrometers operating at, or near, room temperature. Such  $HgI_2$  systems offer the promise of being developed into high resolution, elemental analysis instruments whose performance can equal that of spectrometers based upon cryogenically cooled silicon and germanium. Many space instruments, such as the Scanning Electron Microscope and Particle Analyzer (SEMPA) currently being developed by Jet Propulsion Laboratory (JPL), to be flown on the Mariner Mark II comet mission, can greatly benefit from the use of a  $HgI_2$  x-ray spectrometer.

As a result of collaboration between the USC Institute of Physics technical staff and JPL, it was demonstrated that  $HgI_2$  detectors can be employed in a high resolution x-ray spectrometer, operating in a scanning electron microscope. An energy resolution of under 200 eV has been achieved for a system that was set up external to USC (at JPL).

Based upon the encouraging results obtained to date on this program, we expect to see continued future progress in the areas of energy resolution, more optimal detector encapsulation, and sensitivity to low energy x-rays.

A second part of the project, carried out in collaboration with the Laboratory of Astrophysics and Space Research of the University of Chicago, concerned the development of  $HgI_2$  x-ray detectors to augment alpha backscattering spectrometers. These combination instruments allow for the identification of all chemical elements, with the possible exception of hydrogen, and their respective concentrations.

Additionally, we report on further investigations of questions regarding radiation damage effects in the  $HgI_2$  detectors. This includes joint studies between USC and the University of Chicago, which build upon the encouraging initial results obtained from earlier irradiation tests using high energy protons.

In the succeeding sections we report upon the following topics:

- \* Basic development of detectors and low noise amplification electronics (including energy resolution enhancement, detector fabrication studies for improved electrical properties, input FET selection, reduction of FET operating temperature, detector surface passivation and encapsulation, and longevity under high vacuum and temperature cycling testing).
- \* Continued development of the x-ray spectrometer for SEMP A space applications.
- \* Development of x-ray and alpha backscattering spectrometers for space applications.

- \* Continued investigation of detector radiation damage effects using high energy protons.

## II. RESULTS

### A. Basic Development of Detector and Low Noise Amplification Electronics.

#### 1. Further Improvement of Energy Resolution.

##### a. Background

A key element in x-ray spectroscopy systems, particularly for arrays of light elements, is the quality of the energy dispersive detector. For such analyses, a system energy resolution on the order of 200 eV (FWHM) is required. To date, this ordinarily has required the use of cryogenically cooled silicon or germanium x-ray detectors. In some cases, proportional counters also can be used, and it was specifically to compete with these devices that our first mercuric iodide detector studies were addressed. As  $\text{HgI}_2$  detector performance steadily improved, however, this goal has been changed from proportional counter performance to that of a cryogenically cooled silicon detector.

Work done at the University of Southern California has shown that the energy resolution for a mercuric iodide ( $\text{HgI}_2$ ) spectrometer can approach that of silicon or germanium spectrometers. A major advantage to the use of these  $\text{HgI}_2$  systems is that they do not require liquid nitrogen for cooling. Over the past few years, total  $\text{HgI}_2$  system resolutions have been progressively lowered from initial values of 750 eV or more, to 300eV (FWHM) for the 5.9 keV  $\text{Mn-K}_{\alpha}$  line, when both the detector and its preamplifier were operated at room temperature, or slightly below. The resolution, measured for the 1.25 keV  $\text{Mg-K}$  line, was 245 eV (FWHM) (1-3).

More recently, though, even better (lower) resolutions, to 175 eV (FWHM) for the 1.5 keV  $\text{Al K}_{\alpha}$  line, have been obtained when mercuric iodide detectors were coupled to low noise electronics (4). For that result, moderate cooling, obtained from a small thermoelectric cooler, was used on the preamplifier's input field effect transistor (FET) to lower its temperature to approximately -40°C. (A Marlow model MI 3026 miniature 3-stage thermoelectric device was used). The detector was cooled to about 0°C. The resolution with the one-stage Peltier cooler for the 5.9 keV  $\text{Mn-K}_{\alpha}$  line was 225 eV (FWHM).

Even more recently, in collaboration with JPL, a best-ever, total system resolution of 198 eV (FWHM) was obtained for the 5.9 keV  $\text{K}_{\alpha}$  line of Mn, using a thermoelectrically cooled system which was installed in the Scanning Electron Microscope and Particle Analyzer (SEMPA) instrument. The noise contribution for the thermoelectrically cooled electronics was about 152 eV (FWHM). This result, published in Reference 5, is presented in Figure 1. It should be noted that this high energy resolution result has been obtained not only at the USC laboratories, but also outside, in tests made at JPL.

The foregoing thus represents the achievement of a major milestone, and confirms the general feasibility of the use of  $\text{HgI}_2$  detectors for high resolution spectroscopy applications in space instrumentation (6-8).

### b. Detector Fabrication

It has been noticed that the interface between an evaporated palladium contact and the  $\text{HgI}_2$  crystal is a source of excess electronic noise. We have conducted several tests with different  $\text{HgI}_2$  crystals in order to help understand and clarify this effect.

Several detectors were fabricated using identical processing and handling techniques. Crystal thicknesses and electrical contact areas were kept constant. The detectors also were tested under identical conditions, using the pulser method for determining the electronic noise contribution. These detectors showed variations in their electronic noise of about 100 eV (FWHM). These variations could not be accounted for by the small differences in their leakage currents, so some other factor was suspected as the cause.

Figure 2 shows the equivalent circuit of the detector-preamplifier system.  $R_s$  represents the equivalent series resistance of the contact-crystal interface. Variations in  $R_s$  may significantly change the detector noise performance.

Calculations that have been done using Equation (1) from Reference 1 show that the experimental noise values are much higher than would be expected. This discrepancy can be attributed to the added noise resulting from a contact having additional equivalent series resistance. This effect is particularly important for the case of short shaping times, where the predominant noise contribution does come from the equivalent series resistance.

The contributing factors to the total equivalent series noise resistance are shown in the following equation:

$$R_s' = R_s + R_{sd}(C_d/C_{in})^2,$$

where :

$R_s'$  = total equivalent series noise resistance, ohms

$R_s$  = FET equivalent series noise resistance, ohms

$R_{sd}$  = detector equivalent series noise resistance, ohms

$C_d$  = detector capacitance, farads

$C_{in}$  = total input capacitance of the detector and preamplifier, farads

By subtracting the FET equivalent series noise, it is possible to establish the contribution from the contact-crystal equivalent series resistance. We made use this method for testing and characterizing different interfaces on  $\text{HgI}_2$  crystals. In order to change the crystal-contact interface conditions, we used several different detector preparation and processing methods to attempt to modify and control and minimize the value of  $R_{sd}$ :

- \* Vary the concentration of the etching solution (from 1% to 30%).
- \* Expose the detector surface to different atmospheres, including dry air, wet air,  $\text{N}_2$ , Ar, prior to evaporation of the metal contact.

- \* Examine different contact metals.
- \* Investigate the use of an intermediate layer between the  $\text{HgI}_2$  surface and the metal contact layer.

Some improvements have been observed with these changes, and further investigations will be made. Final results will be reported at a later time.

#### c. FET Selection

We have been able to achieve significant improvements in the electronic noise by experimenting with newly developed FET structures made by the Interfet Corporation. One new FET, type SNJ14L03, has a geometry which produces a better figure of merit (i.e., the ratio of transconductance to input capacitance,  $g_m/C_i$ ) for this application. Using this FET, the electronic noise was reduced by about 25 eV for 6.1 pF capacitance  $\text{HgI}_2$  detectors. In the SEMPA system operating at JPL, changing the FET from a 2N4416 to an SNJ14L03 reduced the noise from 175 eV (FWHM) to 152 eV (FWHM). Energy resolution for the  $\text{Mn K}_\alpha$  peak was accordingly improved, from 225 eV (FWHM) to 198 eV (FWHM).

#### d. Reduction of the FET Operating Temperature

Optimal FET performance in detector preamplifier applications is achieved when the operating temperature is maintained at about 120°K-140°K. Although this cannot be achieved with our three stage Peltier cooler, it is nevertheless important to minimize the temperature that is obtained. The main part of the expected improvement will come from the reduced series noise of the FET, because of the explicit dependence of this term on temperature, and also because the transconductance of the FET increases with decreasing temperature. An additional decrease in noise can be expected because lowering the temperature decreases the gate current of the FET. In order to lower the temperature of the FET as much as possible, we have continued work on a project to optimize the FET/Peltier cooler thermal system.

One key aspect which received careful examination was the thermal design of the FET's support structure. Collaborating researchers at JPL made a detailed computerized thermal analysis of our present design. As a result, we were able to identify the main sources of thermal loading of the Peltier cooling. Decreasing the thermal conductive load presented by the FET support structure, and replacing the direct LED pulsed light input with an optical fiber input (for resetting the FET), significantly lowered the total thermal load for the Peltier cooler used in the present design.

### 2. Detector Surface Passivation and Encapsulation.

Proper  $\text{HgI}_2$  surface passivation and device encapsulation are critical for insuring long term detector reliability, an obvious prerequisite for multi-year space flight applications which may sometimes subject the detectors to adverse conditions. Although unprotected mercuric iodide crystals do not exhibit gross short or long term effects when exposed to normal laboratory storage environments, various gases and vapors, including moisture, can adversely affect detector performance. This is evident when one considers, e.g., the effects that surface moisture could have on the leakage currents for these devices, which normally are measured in picoamperes.

One of the most interesting and challenging aspects of this development has been learning how to provide a protective, impermeable, x-ray transparent coating for the  $HgI_2$  that will not degrade the electrical performance, but that will insure the survival of the device during a seven year space mission, such as the Mars Mariner II.

Several promising surface treatments have been identified to date, and although more work is required, it is seems certain that at least one of the candidate encapsulation materials will prove suitable for the long term protection of  $HgI_2$  detectors. The materials which have undergone initial development and testing are silicones, acrylics such as Conap CE-1170 and polymethylmethacrylates (PMMA), and Parylene.

Some room temperature curing silicone compounds that worked well in a laboratory atmosphere provided little protection against  $HgI_2$  evaporation in vacuum, perhaps because the vacuum removed moisture from the compounds.

The coatings which were applied as liquids and which cured by solvent evaporation, such as the acrylics, proved to be chemically compatible and an excellent barrier to evaporation, and appeared to work well in protecting the detector. However, applying an appropriately thin coating to a detector from any solvent based system was found to be impractical for two major reasons: First, most solvent based systems have the problem that  $HgI_2$  is significantly soluble in the solvent. Second, it was difficult to control the thickness of the coating so that, simultaneously, the coating over the active area was thin, while the coating at the edges was thick enough to prevent evaporation. The solubility of the  $HgI_2$  resulted in the applied coatings always containing small amounts of  $HgI_2$  that produce noticeable x-ray absorption. Typically, the difficulty in thickness control resulted in detectors that had significantly attenuated low energy sensitivity due to excess thickness on the active area, but were still poorly coated at the edges.

This x-ray absorption in the coating can readily be seen in the bremsstrahlung spectrum from a pure carbon target excited with electron beam in a scanning electron microscope. Figure 3 shows the superposition of two bremsstrahlung spectra. The absolute heights of the spectra are arbitrary, but the relative low energy performance of each detector can be estimated by comparing their response below 5 keV to that above 5 keV where the attenuation of each is minimal. The upper curve was obtained from an early detector that had no protective coating over the active area, so that the x-ray intensities are not significantly attenuated above 1.8 keV (the peak at 1.78 keV is probably fluorescence of Si-K lines from nearby silicone rubber). The lower curve is the spectrum obtained with a PMMA-coated detector that clearly shows the Hg-M absorption edges as well as significantly attenuated x-ray intensities below about 3 keV. Small absorption edge artifacts are tolerable, but the loss of low energy sensitivity due to  $HgI_2$  and PMMA absorption would limit such a detector's usefulness in analyzing for light elements, such as Na and Mg.

The Parylene coating has several desirable attributes, including its method of application, polymerization and deposition from a vapor, which allows for a very uniform and well controlled coating thickness. Corners and edges are typically coated to the same thickness as are open surface areas. The relatively low atomic number of these coatings make them close to ideal for being relatively transparent windows for x-rays entering the detector.

The most effective coatings tested to date have been such polymers, deposited from the vapor phase. The most extensively studied has been polymerized dichloro-di-

1,4 xylylene (Union Carbide Parylene-C). Tests including storage at elevated temperatures (80 to 100°C), dipping in KI solutions, and many months operation in vacuum have clearly demonstrated that this material is chemically compatible with HgI<sub>2</sub> and a good barrier to HgI<sub>2</sub> diffusion and external corrosive materials.

As a consequence, we have recently obtained a system for Parylene-C deposition, specially designed and built for this purpose at JPL. Figures 4a and 4b show photographs of a custom built Parylene system. The system required some "fine tuning" to minimize exposure of the HgI<sub>2</sub> crystals to the vacuum before initiation of the polymer deposition, and to better control the thickness of the deposited layers. We now are ready to start a program to determine the optimum thickness of the coating. It is estimated that this will be in the range of 1 μm to 4 μm. We will test specially prepared samples using the above described techniques (elevated temperature, KI solution, vacuum). Finally, detectors coated with precisely controlled thicknesses of the coating will be examined in our testing system, under 10<sup>-6</sup> Torr vacuum and thermal cycling, for the long term stability of their spectral response characteristics.

A 2 μm thickness of Parylene-C produces about 10% attenuation of x-rays at 2.8 keV. In order to minimize the attenuation of x-rays at low energies, we considered other Parylene-C alternatives. Polymerized di-para-xylylene (Union Carbide Parylene-N) should have more desirable, low attenuation properties, since it does contain chlorine atoms, with their accompanying high x-ray absorption. However, this material has only undergone preliminary evaluation by us and it may be necessary to use thicker Parylene-N coatings, because of its relatively higher permeability for some materials.

A full description of this work is presented in Reference 5.

## B. Longevity and High Vacuum Operation

As the HgI<sub>2</sub> x-ray detector project progressed, the long-term behavior of the detector became one of the major considerations. Long-term stability is an important criterion in all applications, especially space missions. The duration of the mission (e.g., about seven years for the Mariner Mark II) places severe demands on the spectrometer, particularly on the HgI<sub>2</sub> detector itself. The system probably will not be operating most of the time, particularly during transit to the target, except for possible periods of test and calibration. This consideration led our group to remeasure HgI<sub>2</sub> x-ray detectors that had been fabricated as long as seven years previously, in the very early stages of HgI<sub>2</sub> development. These HgI<sub>2</sub> detectors had not been stored under any controlled conditions, but were simply kept in plastic boxes in the laboratory.

We found that energy resolution tended to improve slightly with age and storage time. The results seem to be evidence that there is no internal degradation mechanism at work in the HgI<sub>2</sub> crystal itself, over a time period of seven years.

Work on a laboratory research prototype version of the SEMPA instrument at JPL, and other space mission applications, provided the need for development of HgI<sub>2</sub> x-ray detectors compatible with a vacuum environment. Unprotected HgI<sub>2</sub> crystals are not compatible with long term operation under vacuum conditions, and so must be coated or otherwise sealed for such an operation, as was described in the previous section.

For comprehensive testing of detectors and encapsulants under vacuum and/or thermal cycling conditions, a special apparatus with four separate detector chambers has been constructed. Each chamber houses a detector and input field effect transistor,

placed on separate thermoelectric coolers. All test chambers are connected to a common turbomolecular and ion pump manifold to achieve vacuums to  $< 10^{-6}$  Torr, or equivalent to conditions in the SEMPA instrument. Figure 5 shows the photograph of the described system.

For the past several years we have been testing detectors encapsulated with Parylene-C, applied by a commercial source, for longevity under conditions of high vacuum ( $< 10^{-6}$  Torr), with temperature cycling (to simulate conditions anticipated for the SEMPA instrument during the Mariner Mark II mission). An  $\text{HgI}_2$  system is also undergoing continuous testing in the SEMPA instrument at JPL.

We have been monitoring the detectors' stability performance by measuring their energy resolution for the  $\text{Mn K}_\alpha$  line, the electronic noise (pulser width) and their peak-to-background ratio. There has been no noticeable changes in the four tested detectors. Certain variations in the results, which are above statistical error, we attribute to changes of the ambient temperature and the lack of stabilization of this parameter in our present system.

Recently we finished also temperature cycling experiment performed on two detectors in the four detector system described above. Two chambers were provided with external heaters in order to keep the temperature of the chamber body at about  $40^\circ\text{C}$ . This feature is also shown in Figure 5. Detectors were cooled with Peltier elements to the temperature of about  $-20^\circ\text{C}$ . Simple programmable timer was switching power to the Peltier coolers, allowing for very rapid detector temperature change, between  $-20^\circ\text{C}$  and  $+40^\circ\text{C}$ . These particular temperature limits were chosen in order to match the anticipated temperature extremes which will be experienced by the base plate on the Mariner Mark II mission. The results of this experiment, which was carried to 300 cycles, are presented in Table I. Three hundred full temperature cycles is believed to exceed the number of anticipated temperature changes during the Mariner Mark II mission. There is no noticeable changes in the detectors' performance.

The results of this experiment confirm the good encapsulation properties of Parylene-C, and also verify our technique for detector mounting on the ceramic substrate. Temperature and mechanical shocks did not stress the crystal to such an extent as to generate changes in its electrical characteristics.

#### C. Continuation of the Development of the X-ray Spectrometer for SEMPA Space Applications.

A miniaturized Scanning Electron Microscope and Particle Analyzer (SEMPA) is under development by the Jet Propulsion Laboratory for use on the Mariner Mark II Comet Rendezvous Asteroid Flyby (CRAF) mission, scheduled for launch in 1993. This instrument is designed to image individual dust grains with a resolution of 40 nm and be capable of x-ray analysis of individual grains, on a sub-micron scale, with an energy resolution of 200 eV at 5.9 keV.

The requirements of an interplanetary mission place severe constraints on the selection of analytical instruments, including the choice of an x-ray detector for SEMPA. The use of a Si(Li) detector would require the use of a costly radiative cooler to achieve the required near liquid nitrogen temperatures. There is evidence that a Si(Li) detector system can be operated at degraded performance level with thermoelectrical cooling, however high electrical power and heat dissipation capacity would be needed. Therefore, the use of a  $\text{HgI}_2$  detector was identified as a good choice to minimize power consumption and weight of the SEMPA instrument since the

preamplifier input FET and detector would clearly need to be cooled using only small thermoelectric coolers.

The HgI<sub>2</sub> detectors which have been under development at USC offer substantial advantages in terms of decreased power requirements and fewer thermal/mechanical design difficulties, while still approaching the performance of the cryogenically cooled Si(Li) units.

While not all of the required final goals for the instrument have been achieved yet, tests of the HgI<sub>2</sub> systems were quite successful, and have showed a steady, consistent improvement in overall performance, as the result of carefully addressing the issues of geometric configuration in the SEM, compact packaging that includes separate thermoelectric coolers for the detector and FET, x-ray transparent hermetic encapsulation and electrical contacts, and a clean vacuum environment. By focusing efforts on these specifics, a promising trend of improvement in x-ray performance has been achieved. At this time, the feasibility of an HgI<sub>2</sub> x-ray detector having 200 eV (FWHM) resolution has been demonstrated.

Specific areas which were addressed during this program included the following:

- \* Improving the construction of the HgI<sub>2</sub> x-ray probe to include better thermal design for the FET cooler and its heat sinking the scanning microscope body.
- \* Extending the probe sensitivity to lower energy x-rays by optimization of the beryllium electron backscatter shield and by optimization of detector encapsulation, as described in section III.A.2, above.
- \* Testing advances in the detector and low noise electronics (described in the previous sections) outside of our own laboratory, in particular, in the specific environment of a scanning electron microscope.

#### D. Development of Mercuric Iodide X-ray and Alpha Backscattering Spectrometers for Space Application.

The Laboratory for Astrophysics and Space Research of the University of Chicago, under NASA auspices, has been developing an instrument for obtaining the chemical composition of remote planetary bodies. This instrument is based on three interactions which occur when alpha particles from a radioactive source are incident upon sample matter:

- \* Elastic scattering of alpha particles by nuclei (alpha mode)
- \* ( $\alpha, p$ ) nuclear reactions with certain light elements (proton mode), and
- \* excitation of the atomic structure and subsequent emission of characteristic x-rays (x-ray mode).

This instrument has a long history in space applications. The first two modes have been used in the past to obtain the first detailed chemical analysis of the lunar surface during the Surveyor missions to the Moon in 1967-68. Since then, the instrument has been improved and miniaturized substantially. One substantial improvement was the addition of the x-ray mode to the alpha and proton modes. It was realized that a significant amount analytical information from the instrument was being lost by not utilizing the x-rays emitted from the analyzed sample.

As it turns out, the same radioactive alpha particle source that is used for the alpha and proton modes (in this case  $^{242}\text{Cm}$  or  $^{244}\text{Cm}$ ) is also a very effective x-ray excitation source. This excitation is caused by both the alpha particles themselves (more effective for lower Z elements) and by the L-series x-rays of the radioactive source, which are very effective for the higher Z elements.

The inclusion of the x-ray spectroscopy mode improved the overall performance of the Alpha particle instrument by increasing its accuracy and by extending the sensitivity for some important elements down to the ppm range.

In the past, for the x-ray mode, cryogenically cooled Ge or Si(Li) x-ray detectors have been considered for flight instruments. For some specific cases, where it is applicable, the requirements for cryogenic operation is not a problem. For example, in the proposal for an alpha particle experiment on the penetrant for the CRAF mission, the whole penetrant would have been buried under the surface of the comet, and that would provide the cooling required for the Si(Li) x-ray detector.

Another specific case, in which the University of Chicago was involved, was an alpha backscattering instrument for experiments on board the two Soviet Phobos spacecrafts to provide the chemical composition of the Martian satellite Phobos (and perhaps Deimos). This instrument is based on the University of Chicago's Mini-Alpha design and used a Si(Li) x-ray detector for its x-ray mode. This was possible because the nighttime temperature of Phobos' surface is cold enough to be in the operational regime of the Si(Li) detectors.

However, for vast majority of space applications, the prevailing environmental conditions are such that they will preclude the use of cryogenically cooled Ge or Si(Li) x-ray detectors. There, x-ray detectors operable at room, or near room, temperatures are needed.

Mars is a classical case where room temperature x-ray detectors will be needed. The planet has a tenuous atmosphere (pressure about 5-7 Torr, mostly CO<sub>2</sub>, with some argon) that prevents ambient temperatures from ever being cold enough for Ge or Si(Li) detectors.

For arguments identical to those given above for the SEMPA instrument, it is highly desirable for the alpha particle instrument to have a detector system with high resolution, but without the penalty imposed on the instrument by the detector's cryogenic requirements. Mercuric Iodide x-ray detectors offer an alternative solution for high ambient temperature applications that are not amenable to the use of Si(Li) or Ge detectors.

#### E. Continuation of a Program on Radiation Damage of HgI<sub>2</sub> Detectors.

Radiation damage to semiconductor detectors is known to occur during space flight. Over a multi-year mission, the accumulated damage from cosmic rays and their products can result in changes in the operating properties of solid state detectors. In severe cases the detector can be damaged to a point that it stops functioning completely.

There is practically no data in the literature on radiation damage of HgI<sub>2</sub> x-ray detectors. Some very preliminary results have been obtained with gamma detectors by

Becchetti et. al. (9). Recently, we have performed some preliminary tests of mercuric iodide x-ray detectors to assess their vulnerability to such radiation.

For that purpose six medium quality  $\text{HgI}_2$  x-ray detectors were selected and their characteristics (leakage current, FWHM of Fe-55 x-ray line, electronic noise, peak to valley ratio of x-ray line, etc.) were measured before the irradiation. All tests were done with a resistor feedback preamplification system. Electronic noise level and energy resolution were not considered as the highest priorities in those tests. These detectors then were exposed to an external beam of 10.7 MeV protons from the Argonne National Laboratory accelerator, at fluxes up to a  $10^{12}$  protons/cm<sup>2</sup>, to see at what point changes in detector performance could be observed. These irradiations were performed during short periods and represent the worst case in detector damage. Usually during transit in space, the rates of irradiation from cosmic rays are much lower, and the detector is self-annealing during that period.

After the irradiation, the same characteristics were measured again and compared with the data obtained before the radiation. Table II lists parameters measured before and after each detector's irradiation. From these results, it is obvious that all six detectors survived the irradiation without any appreciable change in their performance. The small observed changes can be explained in terms of variations in the test conditions.

In many space applications, the expected accumulated doses can be even higher than the above doses. It is important, therefore, to determine the limits to which  $\text{HgI}_2$  x-ray detectors can resist this radiation damage.

Similar studies, at much higher proton energies, are planned to be done in Europe. For that purpose, we will take advantage of radiation damage studies to be made on high purity Ge detectors which will be used in a Mars Observer gamma ray experiment. Professor Heinrich Wnke of the Max Planck Institute in Germany will allow us to irradiate several of our  $\text{HgI}_2$  x-ray detectors at the same time that the Ge detectors were irradiated in the accelerator, using a 2 GeV proton beam. In this way

we will be better able to evaluate the effect of radiation damage on  $\text{HgI}_2$  x-ray detectors from cosmic rays, over the wide range of energies which are expected during space missions.

### **III. SUMMARY AND CONCLUSIONS**

The data and other developments which are presented in this final report offer proof of the overall feasibility of mercuric iodide x-ray spectrometer detectors for a number of potential space applications. These  $\text{HgI}_2$  systems provide the advantages of small size, light weight, high energy resolution, and freedom from the requirement for cryogenic operating temperatures (as needed by Si(Li) detectors).

Further efforts are required in several areas, however, to develop the system to the level of flight hardware. These include additional work to refine and complete the processing procedures, such as the Parylene encapsulation of the detectors, continued studies to determine radiation damage limits, and ongoing development to improve the specialized low noise, low power electronics.

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Figure 5. Photograph of a special apparatus with four separate detector chambers for longevity testing under conditions of high vacuum and temperature cycling.

Table 1. Detector temperature cycling results.

Table 2. List of detector parameters before and after irradiation.

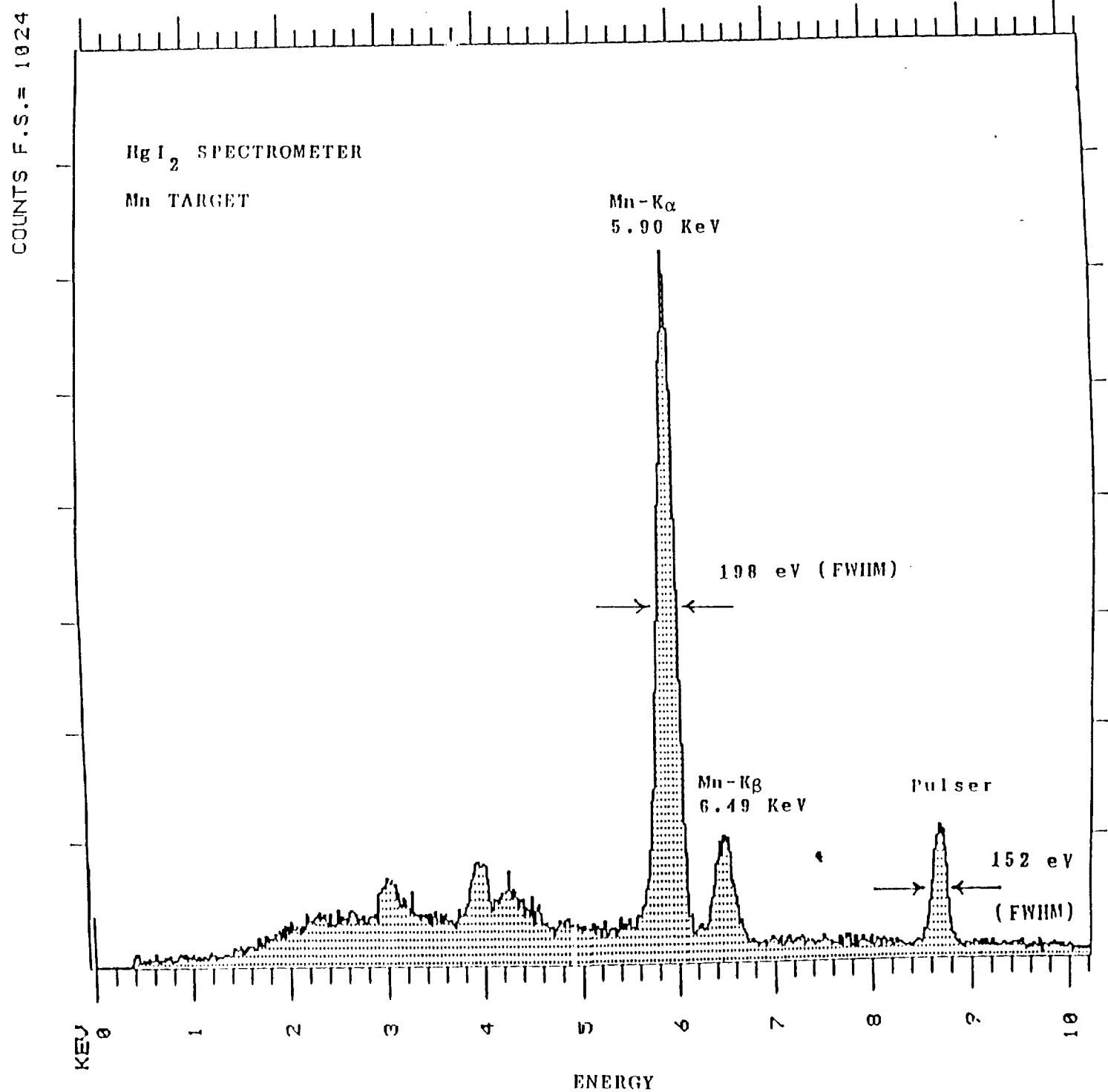
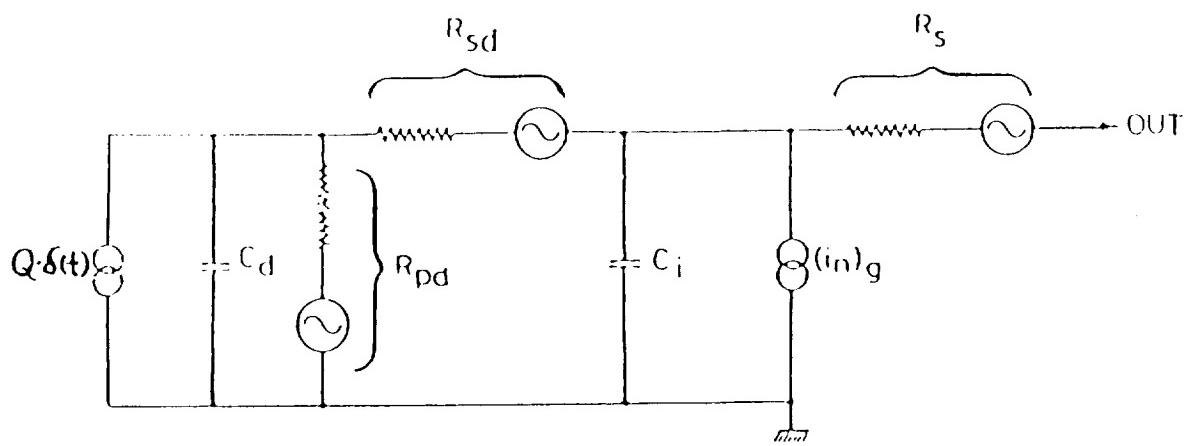


Figure 1. X-ray spectrum obtained with Ug I<sub>2</sub> detector from manganese target



$Q\delta(t)$  = input signal

$(in)_g$  = gate leakage current noise

$R_{pd}$  = detector equivalent parallel noise resistance

$R_{sd}$  = equivalent series noise resistance of the contact-crystal interface

$R_s$  = equivalent resistance of the FET shot noise

$C_d$  = detector capacitance

$C_i$  = preamplifier input capacitance

Figure 2. Equivalent input circuit of the detector-preamplifier system.

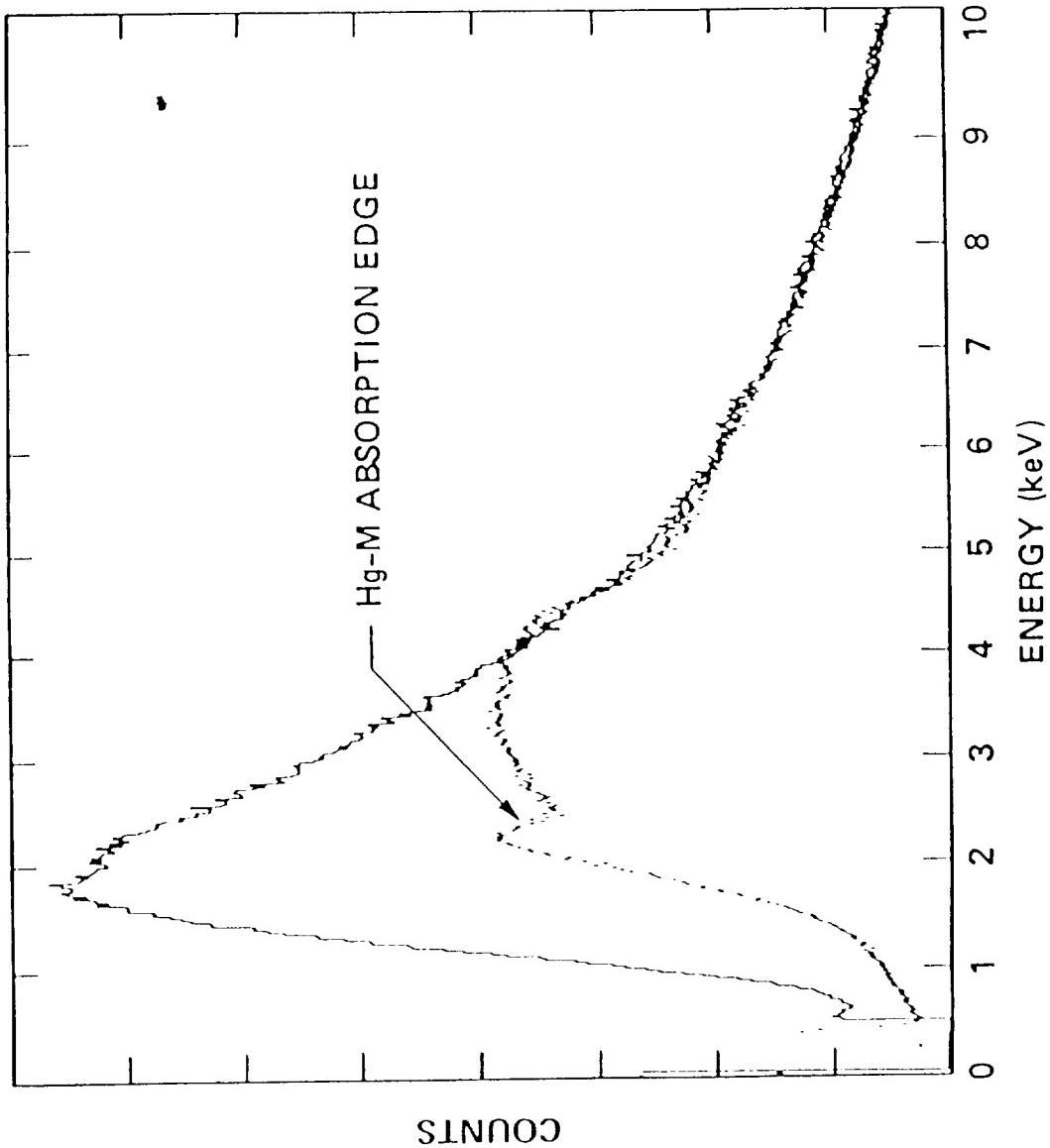


Figure 3: Two x-ray pulse height spectra of bremsstrahlung radiation from carbon targets. The two spectra were obtained with different HgI<sub>6</sub> detectors.

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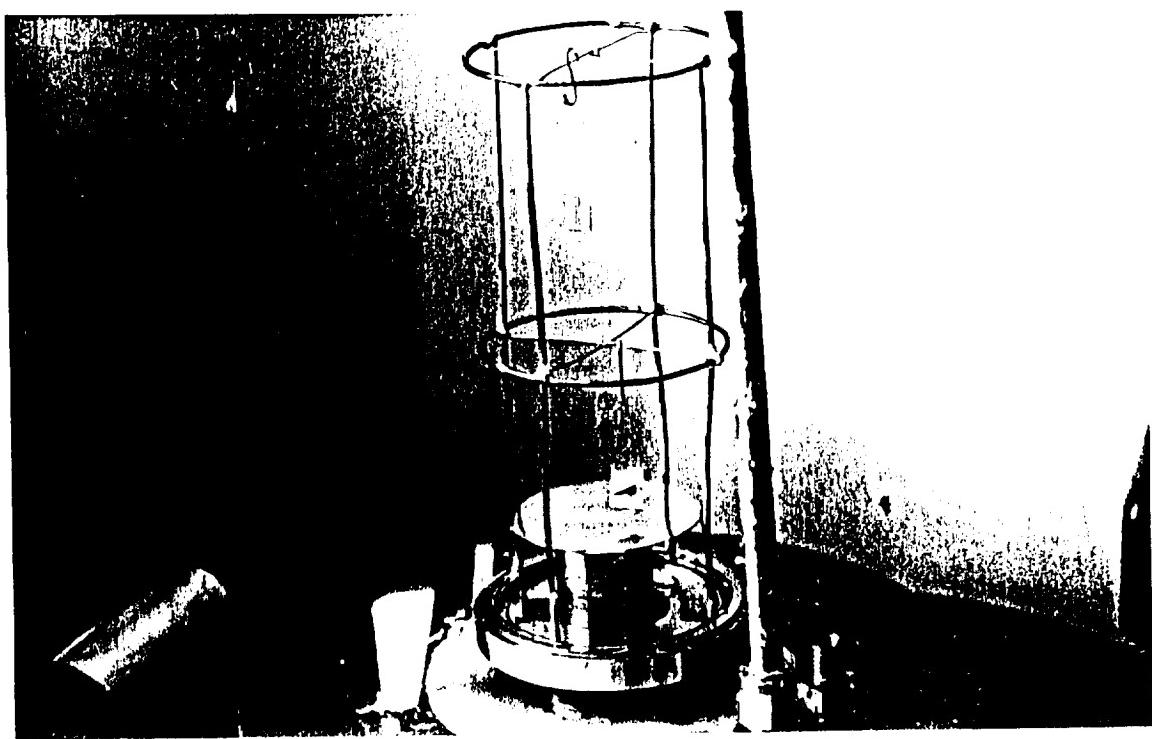
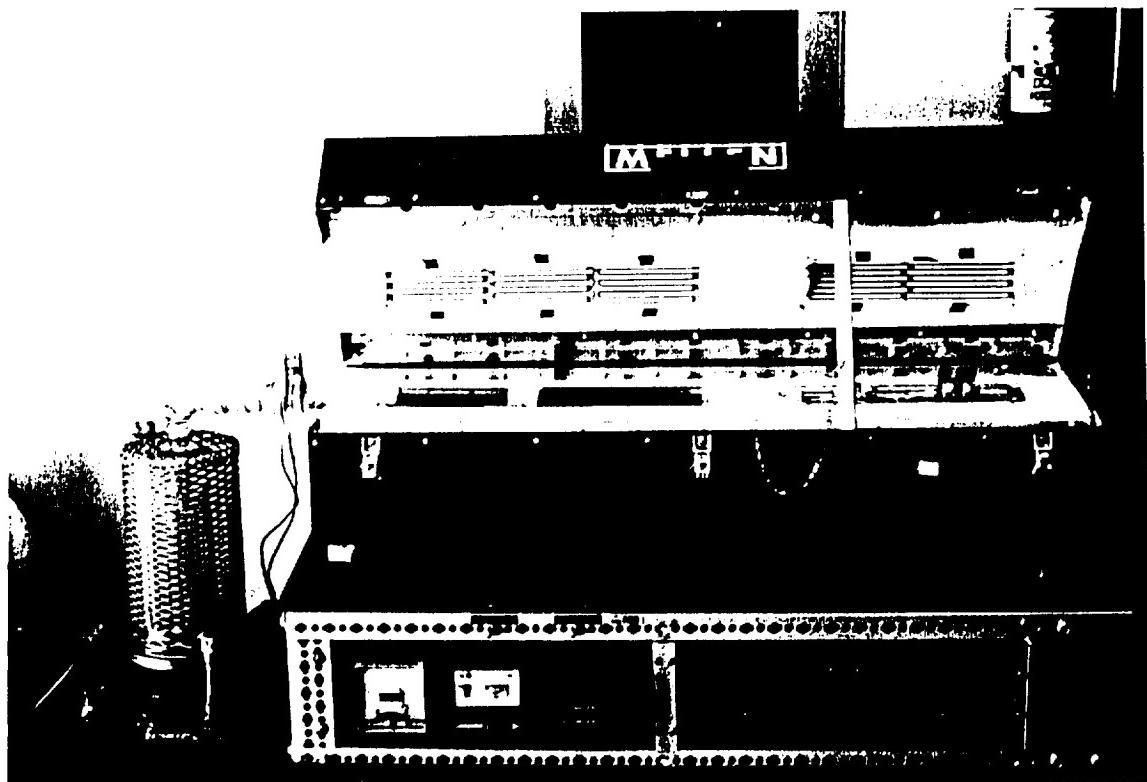


Figure 4. Photographs of custom designed and built system  
for Parylene deposition;  
a) Whole system                  b) Close up of deposition chamber

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Figure 5. A photograph of a special apparatus with four separate detector chambers for longevity under high vacuum and temperature cycling testing

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TABLE I

DETECTOR TEMPRATURE CYCLING RESULTS

DETECTOR N2-1F2

DATE	PESOLUTION (FWHM) IN (eV) PULSER/Mn K /PEAK TO BACKGROUND	# OF CYCLES
08/03/87	296/348/151	BEFORE CYCLING
08/05/87	295/382/153	12 CYS
08/13/87	290/367/154	60 CYS
08/26/87	286/354/151	**150 CYS
09/21/87	291/363/131	300 CYS

DETECTOR N6-2F8

08/03/87	259/298/294	BEFORE CYCLING
08/07/87	258/298/238	2 CYS
08/13/87	248/295/261	5 CYS
08/17/87	248/301/243	10 CYS
08/24/87	243/298/356	18 CYS
09/01/87	249/299/253	**72 CYS
09/14/87	243/292/245	150 CYS
12/02/87	245/303/245	300 CYS

\*\* FULL CYCLE WAS CHANGED FROM 4 HOURS TO 2 HOURS.

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TABLE II

LIST OF DETECTOR PARAMETERS BEFORE  
AND AFTER PROTON IRRADIATION

Det. #	Total dose (protons/cm <sup>2</sup> )	FWHM (eV) before after	FWHM(eV) Pulser	Peak to Background Ratio	Leakage current (pA)
N8-8F2	1.0 <sup>12</sup>	473	437	152:1	0.9
		459	380	220:1	0.8
K7-11DF2	1.0 <sup>12</sup>	532	385	165:1	4.0
		502	394	152:1	3.9
N8-8F2	6.6x10 <sup>9</sup>	394	311	46:1	0.3
		409	370	43:1	1.3
E11-5LF2	6.6x10 <sup>9</sup>	472	366	66:1	4.0
		496	369	67:1	2.3
N12-6F1	4.8x10 <sup>10</sup>	490	422	193:1	3.7
		426	350	248:1	4.8
S8-2SF5	4.8x10 <sup>10</sup>	483	426	215:1	0.2
		491	409	196:1	0.1

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VI. APPENDIX - RECENT TECHNICAL PAPERS

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# **PERFORMANCE AND DURABILITY OF HGI X-RAY DETECTORS FOR SPACE MISSIONS**

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PERFORMANCE AND DURABILITY OF HgI<sub>2</sub> X-RAY DETECTORS FOR SPACE MISSIONS

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Considerable recent progress has been achieved in HgI<sub>2</sub> detector fabrication technology and amplification electronics. An energy resolution of 198 eV (FWHM) has been obtained for the Mn k<sub>α</sub> line of 5.9 keV in a practical x-ray probe without the use of cryogenic cooling. Detectors prepared with Parylene-C encapsulation have demonstrated perfect reliability in two-year tests under high vacuum, and temperature and bias cycling. Other HgI<sub>2</sub> detectors were used to demonstrate proton radiation damage resistance to levels of 10<sup>12</sup> protons/cm<sup>2</sup> at 10.7 MeV.

Introduction

A key element in x-ray spectroscopy systems, particularly for arrays of light elements, is the quality of the energy dispersive detector. A system energy resolution of 140 to 200 eV (FWHM) at 5.9 keV is typically required depending on the application. To date, this ordinarily has required the use of cryogenically cooled silicon or germanium x-ray detectors. Recent work has shown that the energy resolution for a mercuric iodide (HgI<sub>2</sub>) spectrometer can approach that of silicon or germanium spectrometers.

A major advantage to the use of a HgI<sub>2</sub> system in many applications is that it does not require liquid nitrogen for cooling (1-3). It is this advantage that has led to a program to evaluate and develop such a system for the Scanning Electron Microscope and Particle Analyzer (SEMPA) instrument (4,5,8) being developed for NASA's Mariner Mark II Comet Rendezvous/Asteroid Flyby Mission (6). This mission has a planned duration in excess of seven years and thus makes considerable demands on long term reliability of the spacecraft as well as science instruments such as SEMPAs. At the start of the program the ultimate ability of HgI<sub>2</sub> detectors to meet both the SEMPAs resolution requirement (200 eV) and the longevity requirement was quite uncertain.

Significant progress has been achieved in HgI<sub>2</sub> detector performance through improvements both in fabrication technology and low noise amplification electronics. Significantly improved spectral response has been achieved with the introduction of a guard-ring detector contact and a collimating metal shield close in front of the detector. Especially at high count rates, these two advancements have minimized noise contributions from charge generation and collection in regions with weak electric fields (2,7). Reduction in preamplifier electronic noise has been achieved through

utilization of newly developed FET structures made by the Intermet Corporation. One new FET, type SNJ14L03, has a geometry with a better figure of merit (i.e., the ratio of transconductance to input capacitance, g<sub>m</sub>/C<sub>i</sub>) for low capacitance detectors and thus gives lower noise for our application.

Spectral Resolution

The SEMPAs laboratory research prototype, shown in Figure 1, has been a test-bed for the evaluation of improvements to practical detector systems. The x-ray probe system used for ultimate resolution tests is in the lower left part of Figure 1. The details of the probe system are shown schematically in Figure 2. The HgI<sub>2</sub> detector used had an active area of 5 mm<sup>2</sup>.

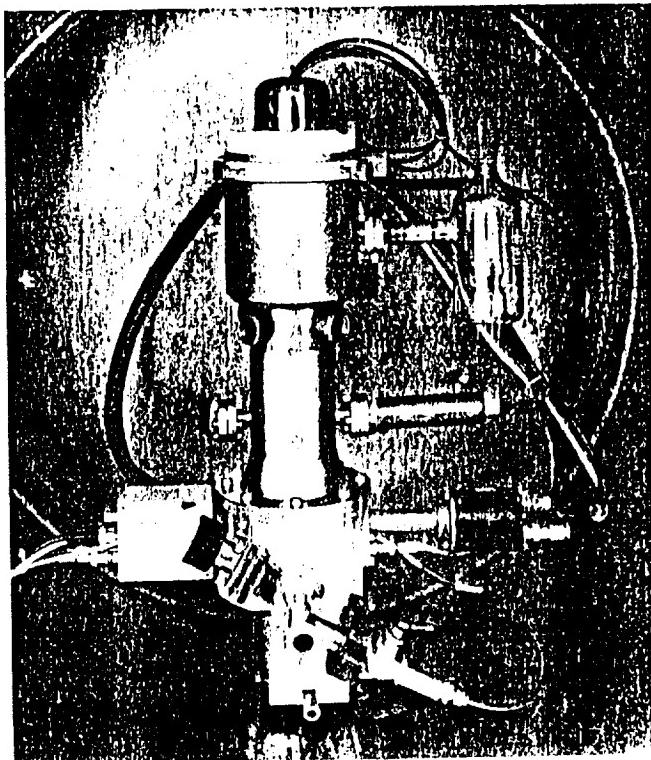


Figure 1. The SEMPAs (Scanning Electron Microscope and Particle Analyzer) laboratory research prototype.

It was mounted 10 mm from the target and intercepted 0.05 steradian solid angle. The detector leakage current was less than 0.1 pA and the detector capacitance was less than 1 pF. The system uses a single stage thermoelectric cooler to reduce the detector temperature to about 0°C. The preamplifier input FET is cooled to about -40°C with a three stage input FET is cooled to about -40°C with a three stage

thermoelectric cooler. The detector is protected from backscattered 15 keV electrons by an 8  $\mu$ m Be window. The detector system is exposed to the vacuum environment of the SEM column. The typical system operating pressure of  $3 \times 10^{-7}$  Torr is achieved by a liquid nitrogen trapped diffusion pump. In this thermoelectrically cooled  $HgI_2$  spectrometer system a

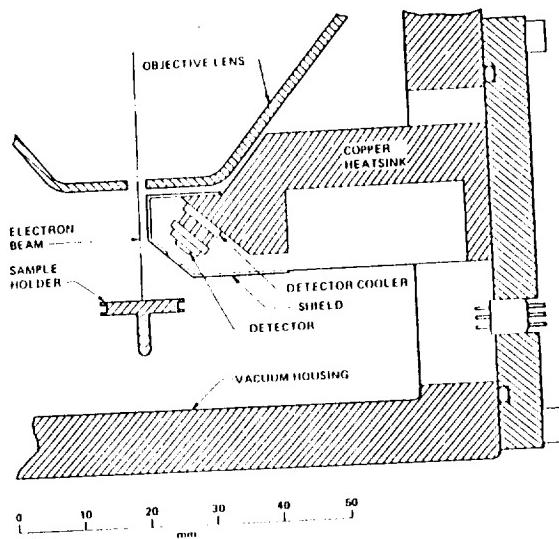


Figure 2. General configuration of the  $HgI_2$  detector in the SEMPA instrument.

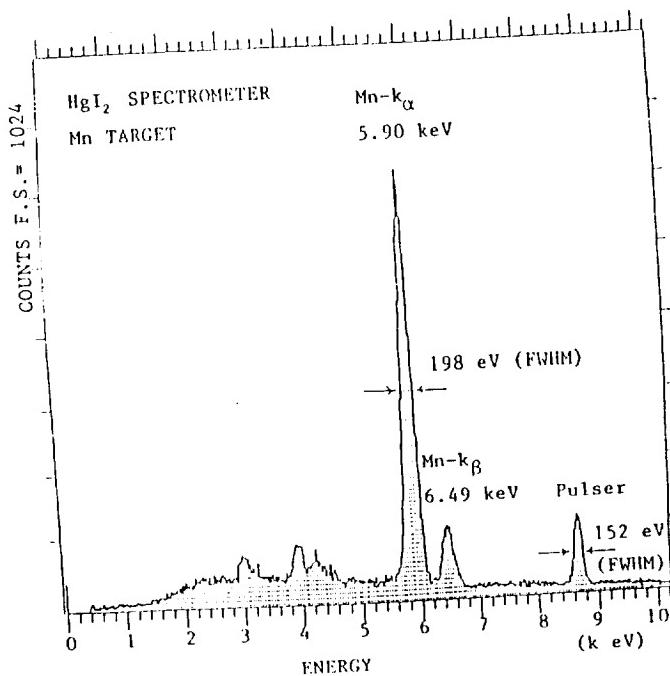


Figure 3. Manganese spectrum taken with the  $HgI_2$  SEMPA prototype detector.

best-ever, total resolution of 198 eV (FWHM) was obtained for the 5.9 keV  $k\alpha$  line of Mn, Figure 3. The electronic noise contribution for this system was about 152 eV (FWHM) measured by pulser method. The main amplifier was a Tennelec TC244. The triangular shaping mode was chosen with the peaking time of 28.8  $\mu$ s.

#### Detector Encapsulants

Proper  $HgI_2$  surface passivation and device encapsulation are critical for insuring long term detector reliability. Although unprotected mercuric iodide crystals do not exhibit gross short or long term effects when exposed to normal laboratory storage environments, various gases, vapors, and particularly a vacuum environment, can rapidly, adversely affect detector performance. Several excellent protective surface treatments have been identified to date. The materials which have undergone initial development and testing are silicones, acrylics such as Conap CE-1170 and polymethylmethacrylate (PMMA), and Parylene (a Union Carbide product).

Some room temperature curing silicone compounds that worked well in a laboratory atmosphere provided little protection against  $HgI_2$  evaporation in the vacuum, perhaps because the vacuum removed moisture from the compounds. Some coatings, especially acrylics, which are applied in a solution and harden with solvent evaporation, have proven to be chemically compatible with  $HgI_2$  and an excellent barrier to  $HgI_2$  evaporation. However, applying an appropriately thin coating to a detector from any solvent based system was found to be impractical for two major reasons: First, most solvent based systems have the problem that  $HgI_2$  is significantly soluble in the solvent. Second, it was difficult to control the thickness of the coating so that, simultaneously, the coating over the active area was thin, while the coating at the edges was thick enough to prevent evaporation. The solubility of the  $HgI_2$  resulted in the applied coatings always containing small amounts of  $HgI_2$  that produce noticeable x-ray absorption. Typically, the difficulty in thickness control resulted in detectors that had significantly reduced low energy sensitivity due to excess coating thicknesses on the active areas, but were still poorly coated at the edges (3).

Parylene coatings have several desirable attributes, including their method of application, polymerization and deposition from a vapor, which allows for a very uniform and well controlled coating thickness even in submicron layers. Corners and edges are typically coated to the same thickness as are open surface areas. The low atomic number of some Parylenes make them useful as x-ray transparent windows. The most extensively tested has been polymerized dichloro-di-1,4-xylylene (Union Carbide Parylene-C). Tests of coating performance have included storage of coated  $HgI_2$  crystals at elevated temperatures ( $75^{\circ}C$  to  $80^{\circ}C$ ), dipping in KI solutions, and many months operation in a vacuum. These test have clearly demonstrated that this material is chemically compatible with  $HgI_2$  and an excellent barrier to  $HgI_2$  diffusion and external corrosive materials.

Initial Parylene-C tests were performed on units whose coatings were applied by vendors, including the Union Carbide Service Center, in San Diego, CA. These initial tests were sufficiently promising to warrant building a small scale coating system for our laboratory that would allow optimization of the coating process and properties for this application. After some initial fine tuning, the system consistently produces water-clear coatings that appear superior to coatings

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which are routinely obtained from commercial vendors. A practical thermal/vacuum process for  $\text{HgI}_2$  crystal coating was developed to minimize exposure of the  $\text{HgI}_2$  crystals to the vacuum before initiation of the polymer deposition. Early coating tests showed that exposure of the  $\text{HgI}_2$  to the vacuum longer than 20 to 25 minutes prior to polymer deposition could lead to degraded final detector performance. A simple laser interferometer system has been developed to help control the thickness of the deposited layers.

As a check on reliability, in addition to visually examining the quality of test coatings under the microscope, tests for pin holes and permeability have been performed using a KI etching solution (potassium iodide solutions are extremely corrosive to  $\text{HgI}_2$ ). A number of detectors with Parylene-C coatings were immersed in a 10% KI solution kept at 75-80°C. The thicknesses of the Parylene coatings ranged from 2 to 4  $\mu\text{m}$ . After about one month, the solution was checked and determined to be over 40% KI, due to evaporation. The crystals were protected by the coating and no effects were found within this period of coating and no effects were found within this period of more than one month. An uncoated crystal would completely dissolve in a few minutes.

Recently we have started experimentation with other Parylenes because of the x-ray attenuation of Parylene-C. The chlorine content of the 2 micron thickness of Parylene-C produces an undesirable 10% attenuation of x-rays at 2.8 keV as well as significantly increasing x-ray attenuation at energies below 2 keV. Parylene-N (polymerized di-para-xylylene) contains no chlorine and produces less x-ray attenuation for a given thickness. Because Parylene-N has significantly different evaporation/polymerization properties than -C, a new deposition process is being developed to produce the optimum protective coatings. Tests similar to that used for evaluating Parylene-C will be used with the new coatings.

#### Longevity Test

The long-term stability of the detectors is an important criterion in all applications, especially space missions. During the CRAF mission, the SEMPA instrument will be operating to perform x-ray analyses only a small fraction of the time. During flight from earth to the comet (several years) the instrument will be idle. However, during the entire mission, the instrument will be under vacuum conditions and exposed to thermal cycles induced by sun exposure and from other nearby science instruments being turned off and on. These storage and cycling conditions led us to a detailed evaluation of the durability of  $\text{HgI}_2$  detectors.

In order to detect any inherent failure mechanisms in the detectors, we have remeasured the characteristics of  $\text{HgI}_2$  x-ray detectors that had been fabricated as long as seven years previously in the very early stages of  $\text{HgI}_2$  development at the University of Southern California (USC). These  $\text{HgI}_2$  detectors had not been stored under any controlled conditions, but were simply kept in plastic boxes in the laboratory. We found that the energy resolutions of all detectors tended to improve slightly with age and storage time. The results seem to be evidence that there is no internal degradation mechanism at work in the  $\text{HgI}_2$  crystal itself, over a time period of seven years. These detectors had all been protected by acrylic and/or silicone coatings. The apparent improvements in performance are probably due to improvements in the electronics used for the measurements.

For comprehensive testing of detectors and encapsulants under vacuum and/or thermal cycling

conditions, a special apparatus with four separate detector chambers was constructed. Each chamber houses a detector and the preamplifier input field effect transistor, each attached to a separate thermoelectric cooler. All of the test chambers are connected to a common turbomolecular and ion pump manifold to achieve clean vacuums to  $10^{-7}$  Torr, or equivalent to conditions in the SEMPA instrument.

For the past two years, four detectors encapsulated with Parylene-C, applied by a commercial source, have been undergoing testing in this system. The coating thicknesses are estimated to be about 4  $\mu\text{m}$ . These detectors have also undergone bias cycling due to power failures at a rate of about once per month. Figure 4 gives a summary of energy resolution tests conducted at the Institute of Physics, USC. The tests on detectors N6-9F7 (in chamber #1) and N3-1F2 (in chamber #2) started in December 1986. The tests on detectors N6-9F8 (in chamber #3) and N3-1F1 (in chamber #4) started in November 1986. We have been monitoring the detectors' stability performance by measuring their energy resolution for the Mn  $\text{K}\alpha$  line, the electronic noise (pulsar width) and their peak-to-background ratios. Curves 1 to 4 in Figure 4 show the energy resolution versus time for detectors N6-9F7, N3-1F2, N6-9F8 and N3-1F1, respectively. There have been no noticeable changes in the parameters of tested detectors. Certain variations in the results, which are greater than expected from statistics, are attributable to changes in the ambient temperature and the lack of stabilization of this parameter in our present system. During a one week short-term test, resolution was observed to vary about 25 eV. This variation could only be caused by system calibration errors, ambient temperature changes, and changes of the amplifier and cooling power supplies between measurements. From Figure 4 we can see that the long-term resolution variations of four detectors were not far beyond these short-term resolution changes, and that there is no clear systematic trend. Tests on these four detectors are still continuing.

Detector N3-1F2 (in chamber #2) and detector N6-9F8 (in chamber #3) were also subject to temperature cycling during part of the two-year test period. Two chambers were provided with external heaters in order to keep the temperature of the chamber body at about 40°C. Detectors were cooled with Peltier elements to about -20°C. A simple on/off programmable timer periodically switched power to the Peltier coolers, causing very rapid detector temperature changes between +40 and -20°C. The intervals of power on and off were from 1 to 2 hours. The actual detector temperatures changed between extremes in 1 to 2 minutes. These temperature extremes were chosen to approximate the limits which will be experienced by the SEMPA base plate during the CRAF mission. The results of this experiment, which was carried to 300 cycles, are presented in Table I. As can be seen from the table, there were no significant changes in the detectors' performance. Three hundred such temperature cycles exceeds the anticipated number of those cycles for the SEMPA instrument during the CRAF mission, and the temperature changes in the mission are expected to be more gradual and, therefore, less stressful for the detector.

The SEMPA x-ray probe system also serves to test the durability and longevity of  $\text{HgI}_2$  detectors used in its routine operations. Routine instrument operations subject the detector to temperature, pressure and bias cycling tests, some of which are unintentional. After over one year of testing, the performance of a  $\text{HgI}_2$  spectrometer is unchanged, showing energy resolution of about 200 eV (FWHM) for the Mn  $\text{K}\alpha$  line. The

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detector is still undergoing continuous testing in the SEMPA instrument at the Jet Propulsion Laboratory (JPL).

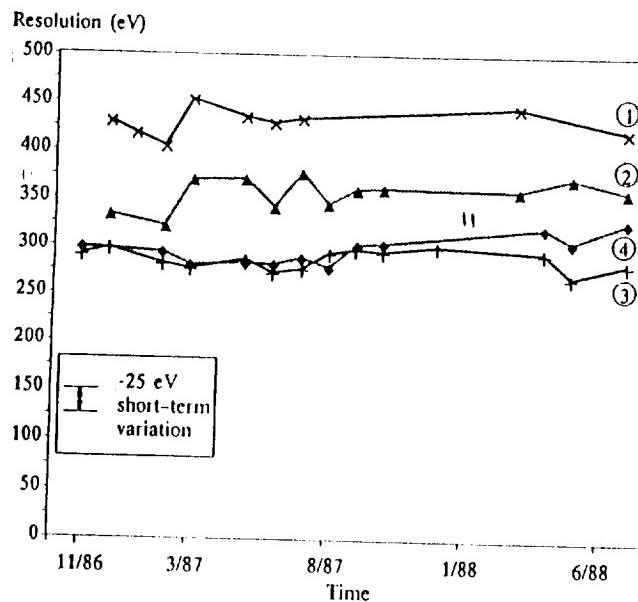


Figure 4. The long-term energy resolution variation for four tested  $\text{HgI}_2$  detectors. The energy resolution (FWHM) was measured for the  $\text{Mn K}\alpha$  line. Curves 1 to 4 correspond to detectors in chambers #1 to #4, respectively.

detector can be damaged to a point that it is no longer useful.

There is little data in the literature on radiation damage of  $\text{HgI}_2$  x-ray detectors. Some very preliminary results were obtained with gamma detectors by Beccetti et al. (9). Recently, we have performed some initial, controlled tests of mercuric iodide x-ray detectors to assess their vulnerability to proton radiation.

For that purpose six medium quality  $\text{HgI}_2$  x-ray detectors protected by PMMA coatings were selected and their characteristics (leakage current, FWHM of Fe-55 x-ray line, electronic noise, peak to valley ratio of x-ray line, etc.) were measured before the irradiation. All tests were done using the same resistor feedback preamplification system. Optimum electronic noise level and energy resolution were not primary considerations in these initial tests. The detectors were exposed to an external beam of 10.7 MeV protons from the Argonne National Laboratory accelerator, to fluences up to  $10^{12}$  protons/cm<sup>2</sup>, to see at what point changes in detector performance could be observed. The fluences were accumulated during periods lasting several minutes and represent the worst case for potential detector damage. Usually during transit in space, the rates of irradiation from cosmic rays are much lower, and the detector may be self-annealing during that period. Within one to two weeks after the irradiations, the same characteristics were measured again. Table II lists parameters measured before and after each detector's irradiation. From these results, it is clear that all six detectors survived the irradiation

TABLE I  
DETECTOR TEMPERATURE CYCLING RESULTS

DATE	RESOLUTION/ PULSER/RATIO*	# OF CYCLES
<b>DETECTOR N3-1F2</b>		
08/03/87	348/296/151	0 CYS
08/05/87	382/295/153	12 CYS
08/13/87	367/290/154	60 CYS
08/26/87	354/286/151	**150 CYS
09/21/87	363/291/131	300 CYS
<b>DETECTOR N6-9F8</b>		
08/03/87	298/259/294	0 CYS
08/07/87	298/258/238	2 CYS
08/13/87	295/248/261	5 CYS
08/17/87	301/248/243	10 CYS
08/24/87	298/243/356	18 CYS
09/01/87	299/249/253	**72 CYS
09/14/87	292/243/245	150 CYS
12/02/87	303/245/245	300 CYS

\* RESOLUTION OF Mn-ka LINE (FWHM) IN (eV)/ PULSER WIDTH (FWHM) IN (eV)/ PEAK TO BACKGROUND RATIO.

\*\* FULL CYCLE WAS CHANGED FROM 4 HOURS TO 2 HOURS.

#### Radiation Damage of $\text{HgI}_2$ Detectors

Radiation damage to semiconductor detectors is known to occur during space flight. Over a multi-year mission, the accumulated damage from cosmic rays and their products can result in changes in the operating properties of solid state detectors. In severe cases the

TABLE II  
LIST OF DETECTOR PARAMETERS BEFORE  
AND AFTER PROTON IRRADIATION

DET. #	DOSE*	RESOLU-	PULSER*	PK/BKG*		LEAKAGE*
				BEFORE	AFTER	
N8-8F3	$6.6 \times 10^9$	394	311	46:1	0.3	
		409	370	43:1	1.3	
E11-5LF3	$6.6 \times 10^9$	472	366	66:1	4.0	
		496	369	67:1	2.3	
N13-6F1	$4.8 \times 10^{10}$	490	422	193:1	2.7	
		426	350	248:1	4.8	
S8-2SF5	$4.8 \times 10^{10}$	483	436	215:1	0.2	
		491	409	196:1	0.1	
N8-8F2	$10^{12}$	473	437	152:1	0.8	
		459	380	220:1	0.8	
K7-11DF2	$10^{12}$	532	385	165:1	4.0	
		502	394	152:1	2.9	

\* Dose: protons/cm<sup>2</sup>  
Resolution: Mn-ka Line (FWHM) in (eV)  
Pulser: pulser with (FWHM) in (eV)  
PK/BKG: peak to background ratio  
Leakage: leakage current (PA).

without any appreciable change in their performance. The small observed changes can be explained in terms of variations in the test conditions.

In some space applications, the expected accumulated doses could be even higher than the above doses. It will be important, therefore, to extend the fluences and energies to which the  $HgI_2$  x-ray detectors are tested for radiation damage susceptibility. Because ten MeV protons represents the low energy range of cosmic rays, it would be extremely interesting to study the effects of irradiating detectors with higher energy protons as well as with other types of radiation that may have different effects.

### Conclusions

All of the experimental and test results described in this paper have shown that  $HgI_2$  detectors are suitable for the ordinary requirements of energy dispersive detectors in x-ray spectroscopy systems. The  $HgI_2$  detectors have shown excellent durability during two-year longevity tests under difficult conditions. Detectors have also shown impressive resistance to proton irradiation damage. Energy resolution measurements better than 200 eV in a practical, noncryogenic detector system represent the achievement of a major milestone, and help confirm the feasibility of utilizing  $HgI_2$  detectors for applications requiring good energy resolution at x-ray energies from 1 to 10 keV.

The research work on improvements in  $HgI_2$  detector fabrication and amplification electronics must continue. We expect to achieve improved low energy detector response by using thinner coatings of protective encapsulants of lower atomic numbers. The long-term life testing will also be repeated with the new coatings. Bias cycling and extended range temperature cycling are planned. Longer exposures and higher proton energies for radiation damage testing are planned in the next phase of this project.

### Acknowledgements

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## ADVANCES IN MERCURIC IODIDE X-RAY DETECTORS AND LOW NOISE PREAMPLIFICATION SYSTEMS

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This paper describes progress achieved at the Institute of Physics in the development of miniature, low power mercuric iodide ( $HgI_2$ ) X-ray spectrometers operating at, or near, room temperature with high energy resolution. The development of single detector spectrometers and, lately, of multidetector array systems has been driven by the specific needs of space exploration and synchrotron radiation applications. Progress has been made in various areas. Advances in the detector fabrication technology, including improved resolution, vacuum operation, improved reliability and longevity, high count rate capability, and the development of soft X-ray transparent encapsulants, are discussed.

### 1. Introduction

Over the past several years, there has been growing interest directed toward the search for room-temperature X-ray detectors capable of high energy resolution. A material found to possess a number of properties that make it quite attractive for such an application is mercuric iodide ( $HgI_2$ ) [1-10]. The broad band gap of this material (2.2 eV) allows for a low detector leakage current at room temperature (typically below 1 pA), and, with a construction designed to keep detector capacitance below 1 pF, the detector electronic noise is very low. Because of the high atomic numbers of its constituents,  $HgI_2$  strongly absorbs X-rays. This feature helps to create a single charge collection situation. When all X-ray interactions take place in the vicinity of the negatively biased entrance electrode, the generated holes travel only minuscule distances, while the more mobile electrons must cross the detector's entire active thickness. In such a case, the contribution of the holes to the induced pulse amplitude in the external amplification circuitry may be neglected. Only the electrons contribute to the spectral response.

Another advantageous property of  $HgI_2$  is its high ionization efficiency. Measuring ionization efficiency as the ratio of the band gap to the mean energy required to produce an electron-hole pair, the value for  $HgI_2$  is (2.2 eV)/(4.2 eV), corresponding to about 52%. This compares very favorably with other materials, e.g. Si, which yields an ionization efficiency of about 30% [11]. Thus, X-rays of equal energy produce a signal only about 13% smaller in  $HgI_2$  than in Si, even though  $HgI_2$ 's band gap is twice as broad. The Fano factor for  $HgI_2$  has been measured to be no more than 0.1. This

value is approximately the same as the experimentally determined value for Si, despite the fact that the trapping phenomena should increase the measured value of the Fano factor of  $HgI_2$  much more than that of Si. Therefore, the limit imposed on energy resolution by the statistical spread in the number of charge carriers produced by an incident radiation event may not be higher in  $HgI_2$  spectrometers than in Si(Li) systems [10].

In the sections below are presented advances in  $HgI_2$  detector fabrication and low noise preamplification systems. Representative spectral responses of  $HgI_2$  spectrometers are shown. Results with improved reliability and longevity under vacuum conditions are presented. A section is devoted to the current status of  $HgI_2$  detector array work. In the last section, a discussion of the applications of  $HgI_2$  technology and plans for future investigations are given.

### 2. Detectors and amplification electronics

The development of single detector spectrometers and, lately, of multidetector array systems has been driven by the specific needs of space applications and synchrotron radiation applications. Advances in the detector fabrication technology include improved resolution, vacuum operation, improved reliability and longevity, high count rate capability, and soft X-ray transparent encapsulants.

Fabrication of the detector proceeds using what have now become standard techniques at USC. Pd electrodes of 100-200 Å thickness are evaporated onto both sides of a slice of  $HgI_2$  single crystal which is typically about

500  $\mu m$  thick. Electrical leads are embedded in the contact areas and the unit is mounted onto a ceramic substrate. BeO has been found to be the most suitable material for this purpose. It has a low dielectric constant and excellent thermal conductivity, thus providing the detector with mechanical support and good thermal contact with cooling elements. In order to passivate the surface and protect the detector from evaporation in a vacuum environment and/or reaction with the ambient, it is sealed with a plastic encapsulant. The protective material must not introduce any additional electronic noise or attenuate incident radiation. Good passivation was achieved with layers of poly-methylmethacrylate (PMMA) delivered from a solvent system. However, these layers varied widely in thickness, tended to crack, and absorbed noticeable traces of  $HgI_2$ . Attention was then turned to the method of vapor deposition. A material found to be quite compatible with this method is poly-dichloro-di-para-xylylene (Union Carbide Parylene-C). Coatings of this material can be made quite uniform and have yielded good results in vacuum work.

Recent additions in detector fabrication have been the introduction of a guard ring, which reduces surface leakage current, and a collimating metal shield. Together, these two elements have been found to significantly improve spectral response at high count rate conditions by minimizing charge generation and collection from regions of weak electric field.

In order to utilize fully the room-temperature capabilities of  $HgI_2$  detectors, ultralow noise preamplifiers without cryogenically cooled FETs are needed. Commercially available equipment does not offer the required parameters. The lowest noise levels are above 500 eV (FWHM) for  $HgI_2$ . The major noise contributors in these systems include excess  $1/f$  noise from the FET chip and its housing, excess stray capacitance and current leakage at the input end, noise originating in the feedback loop elements connected to the front-end FET, and thermal noise in the FET channel. Some of these factors have long been known to arise largely from the FETs' encapsulant materials [2]. This problem has been eliminated by decapsulating the chips and remounting them on ultralow noise materials. The feedback resistor has also been found to be a primary source of noise, and a highly variable one. Noise levels ranged over an order of magnitude for the same value resistors from different manufacturers, depending on variations in construction and handling [7]. While it was found that careful selection of this resistor could reduce noise levels, the best results were obtained by eliminating it altogether and applying the pulsed-light feedback technique. In addition to a lower noise level, this technique also provides high count rate capabilities. Further reduction of noise levels was accomplished by cooling the input FET with the help of miniature thermoelectric (Peltier) coolers. These coolers are very compact (typi-

cally less than 0.5  $cm^3$ ) and use very little power (typically 250 mW) to achieve an effective temperature (-10 to 0°C). These considerations are important for applications that require compact, highly portable units that do not consume much power. Using thermoelectric cooling, typical FWHM noise figures of 180 eV have been achieved. When ultralow noise levels were reached using full-scale, discrete components, attention was turned to the problems of miniaturization and decreasing power consumption. A pulsed-light feedback preamplifier has now been successfully implemented in a unit that measures about  $3 \times 2 \times 0.7 cm^3$  and consumes about 0.22 W of power. The achievement relies on the hybridization technique, which incorporates preamplifier components into a standard 24-pin dual in-line package. The hybrid is designed to have a separate input FET stage, which can be mounted adjacent to the  $HgI_2$  detector and conveniently cooled. Its overall noise performance is as good as that of the full-scale models [13].

Besides the detector and front-end preamplifier, the remaining components of the spectroscopic system used in the experiments were standard, commercially available modules. The main spectroscopic amplifier employed had a long shaping time, usually about 12  $\mu s$ . The amplifier was followed by a commercial multichannel analyzer. The overall configuration was similar to the standard one used in Si(Li) detector systems.

### 3. Spectra

Fig. 1 shows a Mn K (5.9 keV) X-ray spectrum taken with the  $HgI_2$  detector. The solid line represents the spectrum obtained with the detector and input FET at room temperature. The energy resolution is 380 eV (FWHM). The dotted line represents the spectrum obtained with the input FET cooled with liquid nitrogen and the detector operating at room temperature. The energy resolution is 175 eV (FWHM). In both cases the Mn  $K_{\beta}$  peak is clearly visible. The Al K X-ray spectrum, taken with the detector at room temperature and cryogenic cooling of the input FET, is shown in fig. 2. The energy resolution is 145 eV (FWHM). The experimental arrangement used to obtain the spectra presented in figs. 1 and 2 consisted in part of a standard Tracor Xray Inc. Si(Li) liquid-nitrogen cryostat and pulsed-light feedback preamplifier modified to accommodate the  $HgI_2$  detector [10]. The first stage FET was thus at a temperature close to its optimum operating point. The  $HgI_2$  detector was mounted on an alumina ceramic substrate together with a heater and thermistor for temperature control and monitoring. The temperature of the  $HgI_2$  detector was held constant at about 300 K. Although cryogenic cooling of the input FET is not a very practical method of reducing the electronic

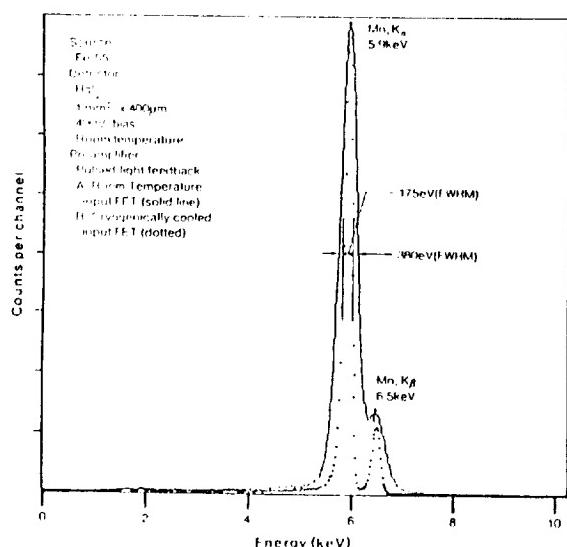


Fig. 1. Plot of manganese K X-ray spectrum. The solid plot was obtained with the detector and preamplifier input FET at room temperature. The dotted plot was obtained with the detector at room temperature and the preamplifier input FET cooled with liquid nitrogen.

noise of a  $HgI_2$  spectrometer, this exercise was useful in revealing the potential of  $HgI_2$  X-ray detectors, and it helped in comparing them with Si[Li] detectors.

Fig. 3 shows a composite of several spectra taken with the Scanning Electron Microscope and Particle Analyzer (SEMPA), which is now being developed at the Jet Propulsion Laboratory (JPL) [14]. The instrument's  $HgI_2$  spectrometer, developed at USC, consisted

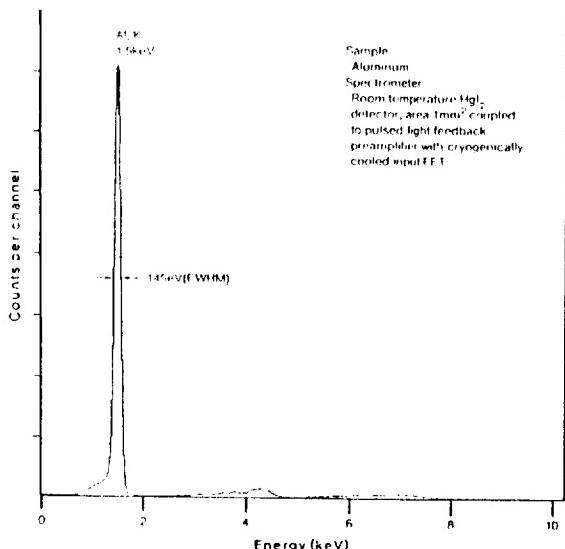


Fig. 2. Aluminum K X-ray spectrum. The detector was at room temperature, and the preamplifier input FET was cryogenically cooled.

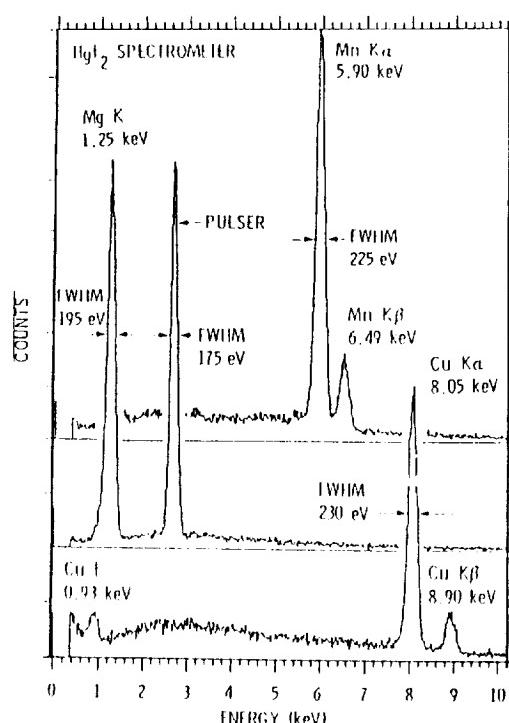


Fig. 3. Composite of spectra obtained with the SEMPMA instrument at JPL.

in part of a detector mounted on a single-stage Marlow M11021 thermoelectric cooler operated at about  $0^\circ\text{C}$ , and an input FET mounted on a three-stage Marlow M13026 thermoelectric cooler operated at about  $-40^\circ\text{C}$ . This temperature is still far from an optimum of  $-120^\circ\text{C}$  for the 2N4416 FET. The electronic noise of the system, as measured by the pulser method, was 175 eV (FWHM). Three lines, Cu L (0.93 keV), Cu K $\alpha$  (8.05 keV), and Cu K $\beta$  (8.90 keV), are clearly visible. The energy resolution of the K $\alpha$  line is 230 eV (FWHM). Also in this figure are the Mn K $\alpha$  and K $\beta$  lines at 5.90 and 6.49 keV respectively. The energy resolution of the Mn K $\alpha$  peak is 225 eV (FWHM). A resolution of 195 eV (FWHM) is obtained for the Mg K line. All spectra shown in fig. 3 exhibit excellent symmetry, background shapes and intensities typical of electron excitation, and neither Hg nor I escape peaks are found (within the acquired counting statistics).

Fig. 4 presents a collection of low-energy spectra showing the K and L lines from a number of elements. The K lines of Al at 1.49 keV, Mg at 1.25 keV, and Na at 1.04 keV were measured. The L lines of Cu at 930 eV and Fe at 705 eV are still clearly resolved from noise. The energy resolution for all of these peaks was approximately 220 eV (FWHM). The overall electronic noise of the system as measured by the broadening of the pulser peak was 185 eV (FWHM) in this case. All peaks displayed symmetrical shapes with little indica-

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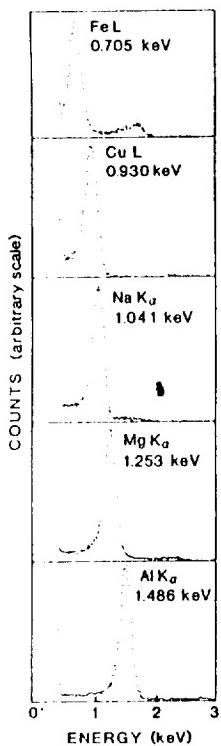


Fig. 4. Composite of low-energy X-ray spectra.

tion of low energy tailing. Fig. 5 shows a spectrum of the K line of oxygen at 523 eV. The extraneous peaks in this spectrum are from impurities on the surface of the piece of beryllium oxide that was used as a target, and which was, in reality, a used detector substrate. The spectra shown in figs. 4 and 5 were obtained with the help of the Van de Graaff accelerator at the California Institute of Technology, which was used to accelerate  $\alpha$ -particles into targets to generate ultrasoft X-rays [15]. The  $HgI_2$  detector had a very thin entrance Pd contact. The detector and the input FET were cooled separately with Peltier coolers. The detector was cooled only slightly, to  $0^\circ C$ , and the input FET was cooled with a two-stage device to about  $-30^\circ C$ .

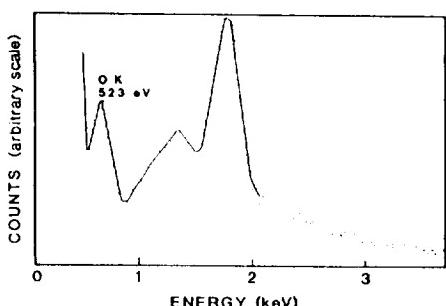


Fig. 5. Spectrum showing the 523 eV peak of oxygen. The other peaks resulted from impurities on the surface of the BeO target.

#### 4. Longevity and high count rate capability

As the  $HgI_2$  X-ray detector project progressed, the long-term behavior of the detector became one of the major considerations. Long-term stability is an important criterion in all applications, especially space missions. The duration of the mission (e.g. about seven years for the Mariner Mark II) places severe demands on the spectrometer, particularly on the  $HgI_2$  detector itself. The system will probably not be operating most of the time, particularly during transit to the target, except for possible calibration and testing. This consideration led our group to remeasure  $HgI_2$  X-ray detectors that had been fabricated as long as seven years previously, in the very early stages of  $HgI_2$  development. These  $HgI_2$  detectors had not been stored under any controlled conditions - simply in plastic boxes in the laboratory. We found that energy resolution seemed to improve slightly with age and storage time. The results seem to be evidence that there is no internal degradation mechanism at work in the  $HgI_2$  crystal itself over a period of seven years.

Work on a laboratory research prototype version of the SEMPA instrument at JPL and other space mission applications provided the need for development of  $HgI_2$  X-ray detectors compatible with the vacuum environment.  $HgI_2$  crystals are not compatible with long-term operation under vacuum conditions but must be coated or otherwise sealed for such an operation. Silicone rubber coatings, which had functioned well in the regular ambient atmosphere, could not stand up to long-term high vacuum conditions. As described above, crystals encapsulated in layers of acrylic or Parylene were provided with good protection against the vacuum as well as good passivation. For the past year, we have been testing detectors thus encapsulated for longevity under conditions of high vacuum ( $< 10^{-6}$  Torr) with temperature cycling (to simulate conditions anticipated for the SEMPA instrument in the Mariner Mark II mission). A  $HgI_2$  system is also undergoing continuous testing in the SEMPA instrument at JPL (details are given in the paper by Bradley et al. in this issue).

High count rate performance of  $HgI_2$  detectors has recently been tested using Stanford's Synchrotron Radiation Laboratory facilities. The introduction of the previously discussed guard ring and collimating shield significantly improved detector performance. Certain electronic baseline instabilities previously observed during intense X-ray illumination [16], which we attributed to charge injection from regions of the detector containing fringe electric fields, have been completely eliminated. The improved  $HgI_2$  detectors were exposed to Cu K X-rays excited by the synchrotron radiation. Tests were performed at up to 200000 counts per second [17,18]. At these count rates, the spectrometer's resolution and throughput were found to be determined by the main

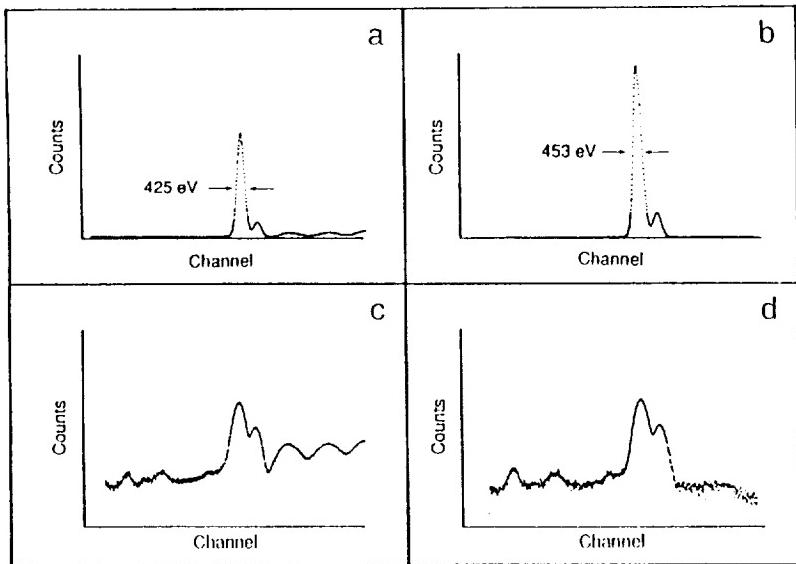


Fig. 6. Linear and semi-log plots of spectra taken with a single  $HgI_2$  detector at 140 kcps input counting rate using two different amplifiers: (a) and (c) Canberra 2020 with 1.5  $\mu s$  shaping time ( $t_s$ ) and no pileup rejection; (b) and (d) Ortec 572 with 2.0  $\mu s$   $t_s$  with pileup rejection.

amplifier's characteristics, in particular, by its shaping time and pileup rejection circuitry. Similar considerations are known to apply in the case of all high resolution solid state spectrometers operated at high count rates.

Representative high count rate results, in linear and log scales, are shown in fig. 6, with  $Cu K_{\alpha}$  and  $K_{\beta}$  lines at 8.04 and 8.94 keV respectively. Input count rates were about 140 kcps. A Canberra 2020 amplifier with 1.5  $\mu s$  shaping time ( $t_s$ ) and without an external pileup gating provision produced figs. 6a and 6c. An Ortec 572 amplifier with 2.0  $\mu s$   $t_s$  and fast pileup inspection circuitry (to gate off the MCA) produced figs. 6b and 6d. Their respective resolutions at 8 keV were 425 eV for the 2020, versus 453 eV for the Ortec. Respective throughputs were 101 kcps and 71 kcps. It would clearly be valuable to investigate the use of amplifier circuitry with an optimized shaping network to improve energy resolution at these high count rates. A design for an amplifier employing a triangular shaping network optimized for such applications has been reported by Goulding et al. [19].

### 5. $HgI_2$ detector arrays

Recently our group has begun to explore the possibility of constructing a  $HgI_2$  X-ray detector array with good energy resolution [20]. Energy dispersive arrays offer the advantages of spatial resolution, large active areas, and high (parallel) count rate capabilities. For such a system we see applications in a variety of fields.

These will be discussed in the next section. Our immediate goal is to develop a submodule consisting of an array of 5–10 detector elements and their preamplifiers. Such submodules could then be organized either linearly or two-dimensionally into larger arrays of 100–400 elements, which, together with signal processing electronics, could be calibrated and coordinated by computer.

Our prototype array spectrometer consisted of five independent channels, separated both physically and electronically. By independently placing the five FET front ends in close, but not intimate, proximity we retained flexibility in identifying and resolving such crosstalk or interference problems as might arise. Five was chosen as a number large enough to be immediately useful scientifically and to display any proximity effects resulting from arraying, yet small enough to be more

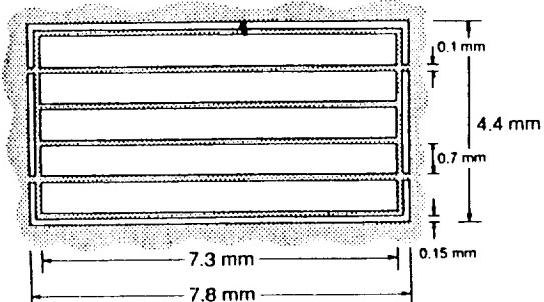


Fig. 7. Drawing of an evaporation mask used to fabricate  $HgI_2$  detector arrays.

readily tractable and less expensive in terms of the costs of electronic processing channels.

Fig. 7 shows a drawing of the mask used to evaporate back contacts onto  $HgI_2$  crystals. The mask allowed five equal-size array elements ( $7.3 \text{ mm} \times 0.7 \text{ mm}$ ) surrounded by a guard ring to be produced. The front contact was achieved by evaporation of a  $7.8 \text{ mm} \times 4.4 \text{ mm}$  Pd electrode. This array was attached to a one-stage Peltier cooler and tested utilizing charge-sensitive, fiber optic pulsed-light feedback preamplifiers. The front end FETs were provided with independent Peltier coolers. In operation, the array was cooled to about  $+5^\circ\text{C}$  and the input FETs to about  $-10^\circ\text{C}$ . An energy resolution of 365 eV (FWHM) was measured. This resolution is only slightly poorer than those previously presented for single  $HgI_2$  detector systems. The electronic noise, measured by the pulser method, was 320 eV (FWHM). The array elements' energy resolution thus seems to be limited principally by electronic noise. We have conducted several tests, floating and grounding elements of the array, which identify the detector contacts (i.e. the Pd- $HgI_2$  interfaces) as the excess noise source, which evidently needs to be eliminated.

Crosstalk measurements were performed to examine any interference problems arising from the close proximity of the preamplification channels. We have found that electronic crosstalk is almost completely eliminated by the introduction of a new fiber optic pulsed light feedback reset technique (invented specifically for this purpose) combined with careful shielding [20]. Charge clouds generated close to the peripheries of two neighboring array elements are subject to lateral diffusion as they are swept across the detector. This results in a partial charge collection by both elements. The charge-collection process in the detector array is schematically

represented in fig. 8. The two partial charges, which sum to the total charge produced, are then recorded as "background" counts in the two elements. In order to minimize background problems of this sort, a stainless steel X-ray shield was employed in front of the detector to block interelement regions from incident X-rays. The low energy backgrounds observed below a single X-ray peak can be used as a good measure of charge division between elements. In a single-element, round detector with well-shielded fringe fields, this peak-to-background ratio (measured as the ratio of 5.9 keV peak amplitude to 3 keV background amplitude) is typically about 300:1, with better devices attaining ratios of 600:1. In the present array elements these ratios were found to be approximately 30:1. We attribute this result to inadequate alignment between the shield and array. It is expected that flat-polishing the front of the detector and repositioning the entrance window's lead wire to facilitate closer physical contact between the shield and the array would significantly improve peak-to-background ratios by reducing interelement charge division problems.

Our current work is directed toward the elimination of charge splitting between adjacent elements and the minimization of electronic noise. Additional effort is directed toward the further miniaturization of the electronic components and the automation of tests under computer control.

## 6. Applications

The unique properties of  $HgI_2$  as a solid state radiation detector compound have by now been well proven and their practical implementation is on its way. In the

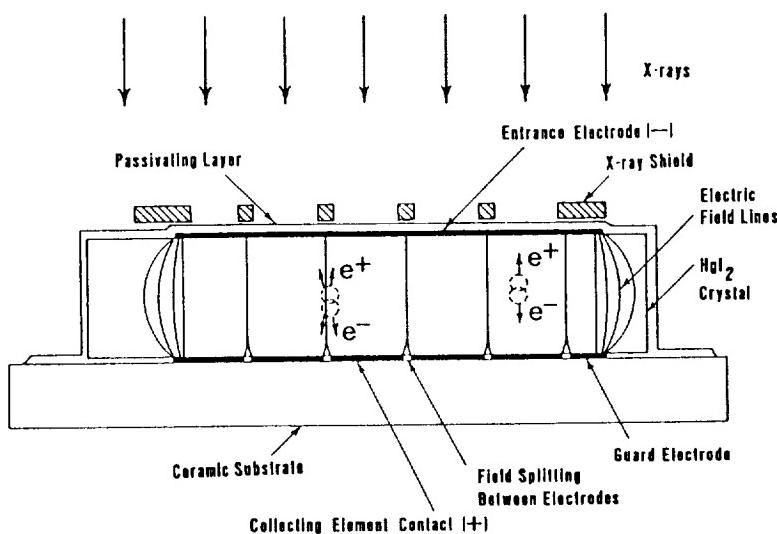


Fig. 8. Cross section schematic of a  $HgI_2$  detector array showing the electric fields and the origin of charge division between electrodes.

X-ray region the energy resolution attainable with  $HgI_2$  detectors at or near room temperature approaches that of cryogenically cooled Si detectors. A prototype  $HgI_2$  X-ray system is operating in the SEMPA instrument at JPL and another at the Stanford Synchrotron Radiation Laboratory. NASA is currently considering use of a  $HgI_2$  spectrometer for the SEMPA instrument in the upcoming Mariner Mark II comet mission.  $HgI_2$ 's room-temperature capability, which eliminates the need for bulky and expensive cryogenic cooling systems, and an electronic design that consumes very little power make such systems very attractive for terrestrial uses as well as for space exploration. The reduced weight and power requirements could reduce the cost of X-ray fluorescence analytical equipment and make available hand-portable instruments for field work. Such instruments could be useful in geological exploration, marine mineral analysis and environmental pollution monitoring. They could also find a place in industrial material quality assurance. Like the SEMPA instrument, earth-bound electron microscopes could incorporate  $HgI_2$  spectrometers with far less stringent design constraints than those imposed by Si(Li) systems, particularly in windowless designs for low energy X-ray work.  $HgI_2$  detectors could be placed directly into the microscope's sample chamber without the problems associated with venting and detector gas poisoning. A number of commercialization efforts have already been initiated to utilize the noncryogenic advantages of  $HgI_2$  technology.

With the development of energy dispersive array detectors,  $HgI_2$  technology is entering a new phase. The detector array opens up new application possibilities. A large active detector area with good energy and spatial resolution would have immediate applications in scanning electron and X-ray microscopy. The ability to handle very high counting rates and intercept fairly large solid angles could allow lower exposure doses to be used for biological or other materials susceptible to radiation damage. The availability of arrays would also significantly benefit a number of areas in synchrotron research, including Extended X-ray Absorption Fine Structure (EXAFS) on dilute solutions, anomalous scattering structural studies and energy dispersive diffraction studies using diamond anvil high pressure cells.

Our continuing efforts will be directed toward improving the energy resolution of  $HgI_2$  spectrometers. This can be achieved with the development of  $HgI_2$  crystal growing methods, the refinement of detector fabrication techniques and the reduction of electronic noise. We will also concentrate on the assurance of reliability and longevity of detectors under adverse conditions. Work on  $HgI_2$  detector array projects will continue with the goals of further refinement in the fabrication procedures, miniaturization of associated components and the automation of tests under computer control.

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## PRACTICAL APPLICATION OF $HgI_2$ DETECTORS TO A SPACE-FLIGHT SCANNING ELECTRON MICROSCOPE

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Mercuric iodide X-ray detectors have been undergoing tests in a prototype scanning electron microscope system being developed for unmanned space-flight. The detector program has met with considerable success, although not all goals have yet been met. This success has been the result of carefully addressing the issues of geometric configuration in the SEM, compact packaging that includes separate thermoelectric coolers for the detector and FET, X-ray transparent hermetic encapsulation and electrical contacts, and a clean vacuum environment.

### 1. Introduction

At the Jet Propulsion Laboratory, the miniaturized Scanning Electron Microscope and Particle Analyzer (SEMPA) [1-3] has been under development for more than eight years. The SEMP A instrument has been selected for the Mariner Mark II (MMII) Comet Rendezvous Asteroid Flyby (CRAF) mission [4,5]. The MMII spacecraft will be launched in 1993, and will rendezvous with comet Tempel 2 in late 1996. The spacecraft will then travel with the comet for about three years while the payload instruments observe the evolution of the nucleus and coma, and determine the nature of the solid and gaseous materials ejected from the nucleus. A penetrator/lander will be implanted in the nucleus to make measurements of its properties. The duration of the mission (about seven years), the distance from the sun (1.49-4.73 AU during science periods), and spacecraft mass limitations place severe constraints on all the spacecraft systems and scientific instruments, and necessitate careful analysis of the performance requirements versus the constraints. The SEMP A instrument has been designed to perform the same functions expected of a laboratory instrument at a somewhat reduced performance level that does not significantly compromise science return. SEMP A will be capable of imaging individual dust grains with a resolution of 40

nm and be capable of X-ray analysis of individual grains on a submicron scale with a resolution of 200 eV at 5.9 keV. The practical X-ray response from 1 to 10 keV and the 15 keV beam energy will permit a quantitative determination of the concentration of elements with atomic number 11 or greater in the range 0.2-100% weight concentration. The entire instrument will have a mass of about 12 kg and consume less than 28 W when in full operation.

Both  $HgI_2$  and Si(Li) X-ray detectors are under consideration for the SEMP A instrument. The Si(Li) detector has the advantage of being a well understood technology, and results have been published that indicate that specially prepared detectors can produce acceptable resolution at temperatures near  $-70^\circ C$  [6,7]. However, studies of one such detector prepared for JPL at Lawrence Berkeley Laboratory suggest that even in detectors with a good resolution at these higher temperatures, the performance changes rapidly with temperature, and temperature stabilization is necessary. On the spacecraft the temperatures needed for the detector and FET would be achieved with thermoelectric (Peltier) coolers. The total input of electrical cooling power needed to achieve the low temperatures for both of these devices is a problem and the thermal/mechanical system design could be difficult and/or expensive. The SEMP A project is planning additional study of the use

of Si(Li) detectors, but the initial work to develop a detector system for SEMPA has been focused on the application of  $HgI_2$  detectors.

## 2. Experimental work

Over the past five years, the laboratory prototype SEMPA instrument has been used to demonstrate a number of technologies that might be appropriate for a flight instrument. The testing of  $HgI_2$  detector systems has been a part of this demonstration program for over three years [8,9]. Although the early systems encountered problems, the recent detector systems have been quite successful. This success has been the result of carefully addressing the issues of geometric configuration in the SEM, compact packaging that includes separate thermoelectric coolers for the detector and FET, X-ray transparent hermetic encapsulation and electrical contacts, and a clean vacuum environment.

The current system configuration is shown in fig. 1. As can be seen, the geometric configurations available for the X-ray system were severely constrained by the working distance of the SEM objective lens (15 mm) and the overall compactness of the vacuum housing. However, a suitable configuration was developed that

beryllium window in front of the detector to protect it from backscattered electrons which would overwhelm the X-ray signal.

The thermoelectric cooling for the  $HgI_2$  detector is provided by a small single stage commercial device. Improved performance above 3 keV was shown to be achieved by cooling the detector to approximately 0°C with about 0.3 A of current. The FET is cooled by a small three-stage device. The FET temperature is lowered to about -40°C with a current of 1.1 A. The total input power is typically 4 W.

Experience has shown that because of the low temperature of the FET and structures in contact with it, cleanliness of the vacuum system is imperative to prevent permanent degradation in the system noise or instability in the SEM operation. The overall system vacuum pumping is provided by a liquid nitrogen trapped diffusion pump. The base pressure in the SEM is stable at about  $2.5 \times 10^{-7}$  T after several days of pumping without venting. Under these vacuum conditions the detector systems have shown no permanent degradation due to contaminants over the longest continuous test intervals (eight months) in the prototype SEMPA instrument. However, a temporary reversible degradation was observed. Tests revealed that, under continuous cooling, the typical system electronic noise

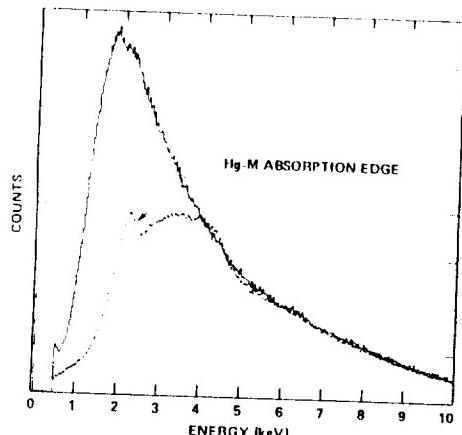


Fig. 2. Two X-ray pulse height spectra of bremsstrahlung radiation from carbon targets. The two spectra were obtained with different  $HgI_2$  detectors.

(PMMA), proved to be chemically compatible and an excellent barrier to evaporation, but applying an appropriately thin coating to a detector from any solvent based system was found to be impractical for two major reasons. First, most solvent-based systems have the problem that  $HgI_2$  is significantly soluble in the solvent. Second, it was difficult to control the thickness of the coating so that, simultaneously, the coating over the active area was thin and the coating at the edges was thick enough to prevent evaporation. The solubility of  $HgI_2$  resulted in the applied coatings always containing small amounts of  $HgI_2$  that produce noticeable X-ray absorption. Typically the difficulty in thickness control resulted in detectors that had significantly attenuated low energy sensitivity due to excess thickness on the active area, but were still poorly coated at the edges.

This X-ray absorption can readily be seen in the bremsstrahlung spectrum from a pure carbon target. Fig. 2 shows the superposition of two bremsstrahlung spectra. The absolute heights of the spectra are arbitrary, but the relative low energy performance of each detector can be estimated by comparing their response below 5 keV to that above 5 keV where the attenuation is minimal. The upper curve was obtained from an early detector that had no protective coating over the active area, so that the X-ray intensities are not significantly attenuated above 1.8 keV (the peak at 1.78 keV is probably fluorescence of Si-K lines from silicone rubber nearby). The lower curve is the spectrum obtained with a PMMA coated detector that clearly shows the Hg-M absorption edges as well as significantly attenuated X-ray intensities below about 3 keV. Small absorption edge artifacts are tolerable, but the loss of low energy sensitivity due to  $HgI_2$  and PMMA absorption would limit the usefulness of such a detector in analyzing for light elements such as Na and Mg.

The most effective coatings tested to date have been

polymers deposited from the vapor phase. The most extensively tested has been polymerized dichloro-di-1,4-xylylene (Union Carbide Parylene-C). Tests including storage at elevated temperatures (80–100 °C), dipping in KI solutions, and many months operation in vacuum have clearly demonstrated that this material is chemically compatible with  $HgI_2$  and a good barrier to  $HgI_2$  diffusion and external corrosive materials. The tests have also shown that the coatings can be produced pin-hole free in micron thick layers if particulates on the detector surfaces are controlled. Parylene has been shown to uniformly coat sharp edges, even razor blade edges. The tests to date have not determined the optimum thickness of the coating, but it is estimated to be in the range of 1–3  $\mu m$ . The only important disadvantage to Parylene-C is the presence of Cl that produces both low energy absorption and an absorption edge. A 2  $\mu m$  thickness produces about a 10% step attenuation at 2.8 keV. Polymerized di-para-xylylene (Union Carbide Parylene-N) has not yet been well evaluated, but it should have most of the desirable properties of Parylene-C with the advantage of not containing chlorine and its accompanying X-ray absorption. However, it may be necessary to use thicker Parylene-N coatings because of its relatively higher permeability to some materials.

The best resolution achieved to date is 198 eV at 5.9 keV with electronic noise of 152 eV [11]. The noise is dominated by the FET. It is expected that improvements in cooling system design and selection of a better FET will yield still better resolution. Special detector fabrication techniques have made operation at higher count rates practical. Rates as high as 2000 counts/s are handled with minimal degradation in resolution or increase in peak tailing.

The key remaining issue to use of a  $HgI_2$  detector in the flight SEMPA instrument is that of proving reproducible long life. We hope to accomplish this through careful detector production control accompanied by systematic testing of devices with processes including accelerated life tests at elevated temperatures, and thermal cycling comparable to that expected on the spacecraft. Recent controlled tests of the potential effects of energetic neutrons and charged particles have shown no appreciable change to  $HgI_2$  X-ray detectors up to  $10^{12}$  protons/cm<sup>2</sup> (10.7 MeV) [11], although some very preliminary tests on gamma-ray detectors showed some effect [10]. We do not expect radiation exposure to limit the useful life of a detector on the CRAF mission.

### 3. Summary

A multi-year program of  $HgI_2$  detector development for the SEMPA instrument has resulted in detector and

detector system designs that show impressively good performance and reliability in the laboratory. A program of additional development and testing, particularly of encapsulation materials and techniques, is underway to establish that 200 eV resolution and un-degraded performance are achievable for a 7-year space mission.

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## DEVELOPMENT OF MERCURIC IODIDE X-RAY AND ALPHA BACKSCATTERING SPECTROMETERS FOR SPACE APPLICATIONS

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An alpha particle instrument in combination with a room temperature mercuric iodide X-ray spectrometer can provide a complete and detailed in situ chemical analyses of extraterrestrial bodies. Conceptual designs of the instrument will be presented. One of the important questions about the resistance of  $HgI_2$  detectors to the radiation damage will be addressed. Our experimental results of exposure of  $HgI_2$  detectors to proton fluxes up to  $10^{12}$  protons/cm $^2$  show no degradation in the detector performance.

The determination of the chemical composition is one of the main goals of every mission to any planetary body of our solar system. The combined alpha particle instrument with its alpha, proton and X-ray modes is

designed to provide remotely a detailed chemical analysis of such bodies.

The alpha particle instrument for space applications is based on three interactions of alpha particles from

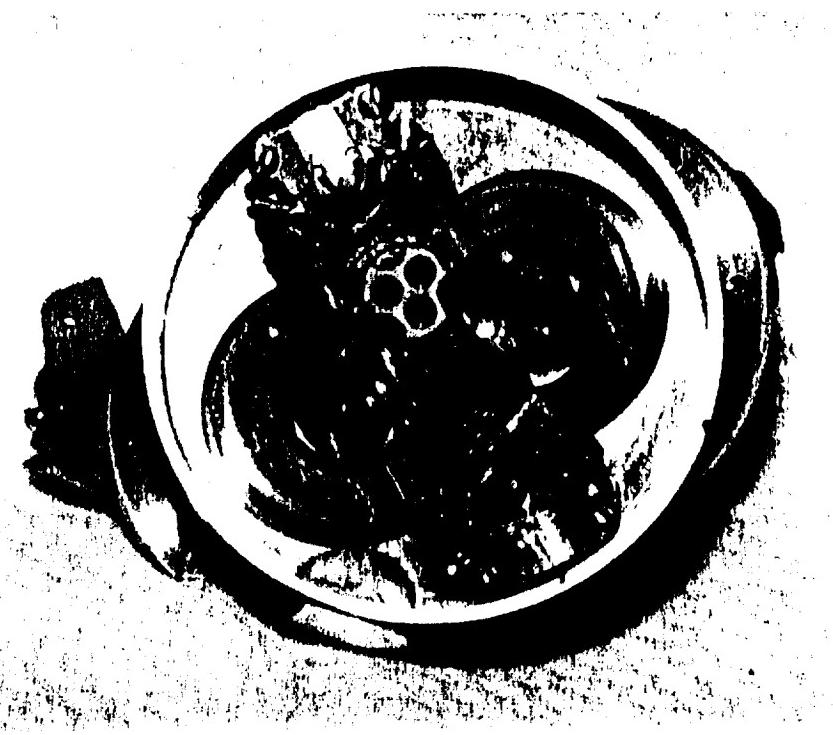


Fig. 1. Photograph of the Minialpha instrument as was proposed for NASA's CRAF mission in 1985.

radioactive sources with matter; elastic scattering of the alpha particles by nuclei (alpha model), ( $\alpha, p$ ) nuclear reaction with certain light elements (proton mode), and excitation of the atomic structure of atoms by alpha particles, leading to emission of characteristic X-rays (X-ray mode). The approach used is to expose material to be analyzed to an alpha radioactive source, and acquire energy spectra of the backscattered alpha particles, protons, and X-rays returned from the sample. The excitation of the atomic structure is provided by the same alpha source used in alpha and proton modes or by an auxiliary X-ray source selected to enhance the sensitivity of the method to certain elements.

An alpha particle instrument with scattered alpha and proton modes provided the first chemical analyses of the lunar surface at three sites during the Surveyor mission of 1967-68 [1]. The results of these analyses were later confirmed by analyses of returned lunar samples. Since 1968 the instrument has been miniaturized in several stages. It has been further demonstrated that such an instrument can identify and determine the amount of all chemical elements (except hydrogen) present in more than about 0.5% by atom in a sample. The instrument and the technique was described in more detail by Economou et al [2]. It was also demonstrated that this instrument can operate under Martian conditions without any degradation in the performance [2,3]. Fig. 1 shows a photograph of the minialpha instrument as was proposed for the CRAF mission. In this case as it was proposed, a penetrator containing the alpha particle instrument with a Si(Li) detector for its X-ray mode, together with many other analytical instruments would penetrate and be buried under the surface of the comet. The temperature inside the comet is very low and the comet itself would provide the cooling for proper operation of the silicon detector.

Similarly, for the X-ray mode of the alpha particle instrument on the Soviet mission in Phobos, which is now being prepared to be launched in July of 1988, a Si(Li) detector is being used due to the fact that the ambient temperature of Phobos during the night is within the operational range of Si(Li) detectors.

However, for many other applications in space (such as Mars for example) where the temperature is much higher, cooled Si(Li) and germanium detectors cannot be used.

The United States as well as the Soviet Union are planning a mission to Mars in the near future (the Soviets just announced a mission to Mars for 1994). Mars has a tenuous atmosphere (about 7 mbar mostly  $CO_2$ , some nitrogen and argon) which prevents the night temperature from getting below the operational range of silicon or germanium detectors. There, room temperature X-ray detectors are needed.

In the past we have considered several semiconductor compounds as room temperature X-ray detectors.

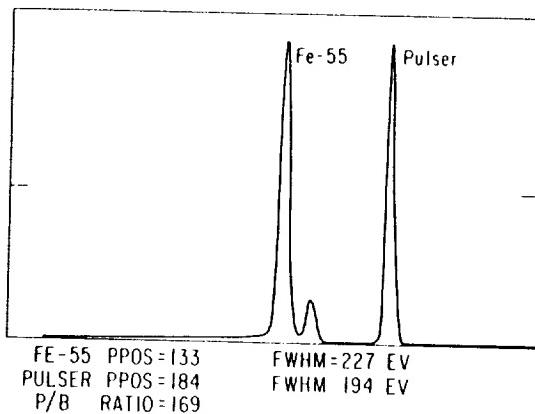


Fig. 2.  $^{55}Fe$  X-ray spectrum obtained with a  $HgI_2$  detector at room temperature and a cooled FET transistor at  $-30^\circ C$ . The spectrum from an electronic pulser is also shown at higher energy.

None of them had the required resolution for a detailed chemical analysis. At that time the goal was to obtain a resolution which would be a little better than the resolution of the gas proportional counter ( $\approx 1.2$  keV at 5.9 keV). The high purity silicon and cadmium telluride detectors showed some promise but they did not have the desired resolution. Mercuric iodide X-ray detectors operating at room temperature seem to provide the greatest promise for space instruments. Developmental work in the recent past done mostly by a group from the University of Southern California [4-7] was very successful in improving the energy resolution of these detectors from about 750 eV at 5.9 keV at that time to the point where now they can almost compete with the cryogenically cooled silicon and germanium detectors. The best resolution now obtainable with  $HgI_2$  X-ray detectors is around 200 eV at 5.9 keV. Fig. 2 shows an  $^{55}Fe$  X-ray spectrum obtained with a  $HgI_2$  detector at room temperature and a cooled FET transistor at  $-30^\circ C$ . The FWHM for  $^{55}Fe$  X-ray line is 227 eV and the electronic resolution is 194 eV. With such a resolution most of the lines from the neighboring elements can be determined. In general high resolution detectors have low background and, as a consequence of this, high sensitivity to minor and trace elements. It has also been shown that  $HgI_2$  detectors can be used for the very low X-ray energy region. The limit has been recently extended down to the oxygen K-line (560 eV) [5].

One of the worries in each space instrument is the radiation damage of the detectors sustained over many years in transit by the cosmic ray radiation. In order to determine detector degradation, six  $HgI_2$  X-ray detectors were subjected to proton fluxes of about 10.7 Mev and up to  $10^{12}$  protons/cm $^2$ . All the detectors survived the radiation. A detailed comparison of the detector characteristics (resolution, leakage current, physical ap-

**Table 1**  
Comparison of detector parameters before and after proton irradiation

Det #	Total dose [protons/cm <sup>2</sup> ]	FWHM [eV] before after	FWHM [eV] pulser	Peak/noise ratio	Leakage current [pA]
N8-8F3	$6.6 \times 10^9$	394 409	311 370	46:1 43:1	0.3 1.3
E11-5LF3	$6.6 \times 10^9$	472 496	366 369	66:1 67:1	4.0 2.3
N13-6F1	$4.8 \times 10^{10}$	490 426	422 350	193:1 248:1	3.7 4.8
S8-2SF5	$4.8 \times 10^{10}$	483 491	436 409	215:1 196:1	0.2 0.1
N8-8F2	$10.00^{12}$	473 489	437 380	152:1 220:1	0.8 0.8
K7-11DE2	$10.00^{12}$	532 502	385 394	165:1 152:1	4.0 3.9

pearance, etc.) showed no significant changes due to the radiation. Table 1 lists the resolution and the leakage currents of these detectors before and after the radiations.

It is our plan to continue these radiation studies with higher proton energies, different kinds of radiation particles and different rates of radiation. But even the present results suggest that the HgI<sub>2</sub> room temperature X-ray detectors can operate in space environments and withstand the expected radiation from cosmic ray bombardment.

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